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# Distribution of Trace Element Emissions from the Liquid Injection Incinerator Combustion Research Facility

Acurex Corp., Jefferson, AR

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The EPA is currently developing regulations on trace element emissions from hazardous waste incineration. However, the data base on trace element emissions from incinerators which can be used to support regulations is very sparse. Data on the effects of waste composition and incinerator operation on trace element emissions are particularly lacking. In response to these data needs, a series of tests was conducted at EPA's Combustion Research Facility (CRF) to investigate the fate of volatile trace elements in liquid injection hazardous waste incineration. In these tests, arsenic in the form of arsenic trioxide ($As_2O_3$) and antimony in the form of antimony trichloride ($SbCl_3$) were added to a methanol base containing varying amounts of chlorobenzene and carbon tetrachloride, and fired in the liquid injection incinerator at the CRF. Test variables included incinerator temperature and excess air level, and feed chlorine content. Test results show a relatively even distribution of both elements between scrubber exit flue gas and scrubber blowdown. Both elements are found in the vapor phase at high temperatures, though most condenses to particulate at scrubber exit temperatures. Designated POHC destruction and removal efficiency (DRE) ranged from 99.99 to 99.999 percent at the afterburner exit to 99.999 to 99.9999 percent in the scrubber exit flue gas. Typical levels of common products of incomplete combustion were measured.

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# DISTRIBUTION OF TRACE ELEMENT EMISSIONS FROM THE LIQUID INJECTION INCINERATOR COMBUSTION RESEARCH FACILITY

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# FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of solid and hazardous wastes. These materials, if improperly dealt with, can threaten both public health and the environment. Abandoned waste sites also have important environmental and public health implications. The Hazardous Waste Engineering Research Laboratory assists in providing an authoritative and defensible engineering basis for assessing and solving these problems. Its products support the policies, programs and regulations of the Environmental Protection Agency, the permitting and other responsibilites of State and local governments and the need of both large and small businesses in handling their wastes responsibly and economically.

This report describes a series of pilot-scale incineration tests performed to investigate the fate of volatile trace elements present in the waste feed of a liquid injection incinerator. In the tests, performed at the U.S. EPA Combustion Research Facility, arsenic and antimony were incorporated into synthetic waste mixtures composed of varying ratios of methanol, chlorobenzene, and carbon tetrachloride. Test variables were incinerator temperature and excess air level, and synthetic waste chlorine content. The report discusses the distribution of these elements fed between flue gas particulate and vapor phases both upstream and downstream of the system's packed tower scrubber, and between the scrubber exit flue gas and blowdown water.

These data are useful to anyone involved in the incineration of hazardous wastes. For further information contact the Alternative Technologies Division of the Hazardous Waste Engineering Research Laboratory.

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## ABSTRACT

The EPA is currently developing regulations on trace element emissions from hazardous waste incineration. However, the data base on trace element emissions from incinerators which can be used to support regulations is very sparse. Data on the effects of waste composition and incinerator operation on trace element emissions are particularly lacking. In response to these data needs, a series of tests was conducted at EPA's Combustion Research Facility (CRF) to investigate the fate of volatile trace elements in liquid injection hazardous waste incineration. In these tests, arsenic in the form of arsenic trioxide ($As_2O_3$) and antimony in the form of antimony trichloride ($SbCL_3$) were added to a methanol base containing varying amounts of chlorobenzene and carbon tetrachloride, and fired in the liquid injection incinerator at the CRF. Test variables included incinerator temperature and excess air level, and feed chlorine content. Test results show a relatively even distribution of both elements between scrubber exit flue gas and scrubber blowdown. Both elements are found in the vapor phase at high temperatures, though most condenses to particulate at scrubber exit temperatures. Designated POHC destruction and removal efficiency (DRE) ranged from 99.99 to 99.999 percent at the afterburner exit to 99.999 to 99.9999 percent in the scrubber exit flue gas. Typical levels of common products of incomplete combustion were measured.

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# SECTION 1

# INTRODUCTION

In support of the EPA Office of Solid Waste (OSW) hazardous waste incinerator regulation development efforts, the EPA Combustion Research Facility (CRF) in Jefferson, Arkansas, conducted a series of incineration tests with hazardous constituent metals, antimony, and arsenic. These metal-emission tests were important because risk assessments show that metal emissions from otherwise properly-operated incinerators can be the largest component of risk to human health and the environment. The tests also studied the formation and emissions of products of incomplete combustion (PICs).

A test burn protocol (1) was submitted in December 1986. This original plan specified incinerating mixtures of No. 2 fuel oil, carbon tetrachloride and chlorobenzene spiked with antimony trichloride and arsenic triioxide. In the actual tests performed, methanol was used instead of the No. 2 fuel oil.

The objectives of the test program were:

- To track the emissions of arsenic and antimony through the incinerator system
- To study the distribution of metals between ash and scrubber water operating conditions and feed chlorine content
- To evaluate whether the metals affect the destruction of other hazardous waste components
- To extend the data base on volatile products of incomplete combustion (PIC) emissions from the incineration of chlorinated hydrocarbons

All tests were performed in the liquid injection incineration system (LIS) at the CRF. This system consists of a pilot-scale liquid injection incinerator which as a main chamber followed by an afterburner chamber. Only the main chamber was fired during these tests. No burner was mounted in the afterburner chamber.

The test protocol called for a series of 18 tests with target conditions selected according to the Box-Wilson factorial design (2) for three parameters varied over five levels. The parameters varied were incinerator temperature, excess oxygen and chlorine content in the waste. Twelve of the 18 tests were completed between January 27 and February 26, 1987. A series of "range-finding" runs preceeded the actual tests to establish the validity

of the sampling and analysis methods, and the typical trace element concentrations in the effluent streams.

This document summarizes the test program in terms of incinerator operation and test results. Section 2 describes the LIS, the waste characteristics, and summarizes the test operation conditions. Section 3 discusses sampling methods, sample recovery, and analysis procedures. Section 4 discusses the test results; test conclusions are summarized in Section 5. Quality assurance and quality control aspects are discussed in Section 6.

# SECTION 2

# FACILITY DESCRIPTION AND OPERATION

As noted in Section 1, the tests were conducted in the LIS at the CRF. The CRF holds a permit to conduct hazardous waste incineration research.

## 2.1 SYSTEM DESCRIPTION

The LIS is capable of firing pumpable and atomizable liquid materials. Table 1 summarizes design characteristics of the incinerator system. Figure 1 shows a simplified schematic of the system, which consists of two combustion chambers, the main- and after-burners. The refractory-lined walls have two layers of insulation with combined thickness of 23 cm (9-in.).

The total primary, afterburner, and transition chamber volume is 5.91 $m^3$ (209 $ft^3$). At design capacity of 586 kW (2 x $10^6$ Btu/hr), the primary combustion chamber has a volumetric heat release rate of 256 $kW/m^3$ (24,690 Btu/hr-$ft^3$). An estimated 4 percent of the input heat escapes the main chamber through the refractory wall. Similarly, about 3 percent escapes the afterburner and transition chambers.

The primary chamber dual-fuel burner is designed to fire propane and atomizable waste liquids. Propane is injected via 12 3.2-mm (1/8-in.) diameter jets located on the gas ring in the burner throat. An oil gun positioned axially in the centerline of the burner consists of a Delavan Model 30615-16 stainless-steel nozzle. It is rated at 11 to 32 ml/s (10 to 30 gph) of No. 2 fuel oil. Steam at 276 kPa (40 psig) atomized the waste liquid.

Propane was fired as needed to achieve the targeted incinerator temperatures. It also enhanced flame stability. A forced-draft fan and a flow-damper controlled combustion air flow. A pitot probe positioned at the throat of a venturi measured the air flowrate. An adjustable air-register imparted swirl to the combustion air. Although secondary air was available, none was used during these tests.

After exiting the afterburner chamber, the combustion gas is cooled in a liquid-sprayed quench chamber to about 80°C (180°F). Then a scrubber packed with 5.1 cm (2-in.) diameter polypropylene Tri-Packs spherical shapes removes HCl and other acid gases in the cooled combustion product gases. Dilute caustic solution neutralizes the acid gases captured in scrubber liquid. A

TABLE 1. DESIGN CHARACTERISTICS OF THE CRF LIQUID INJECTION INCINERATOR

| | | |
|---|---|---|
| **Characteristics of the Main Chamber** | | |
| Length | 3.75m | (12.3 ft) |
| Inside diameter | 0.88m | (2.9 ft) |
| Outside diameter | 1.35m | (4.4 ft) |
| Chamber volume | 2.28 $m^3$ | (81 $ft^3$) |
| Shell construction | 0.63 cm | (0.25 in.) thick cold rolled steel |
| Refractory | | |
| Outside layer | 15.2 cm | (6 in.) thick Kaolite insulating refractory |
| Inside layer | 7.6 cm | (3 in.) thick Kaocrete 1,650°C (3,000°F) high alumina refractory |
| Burner | | |
| Manufacturer | The Engineer Company | |
| | Steam-atomized, liquid flow to 4.5 L/min (1.2 gpm) at 206 kPa (30 psig), pilot gas ignitor | |
| Capacity | 586 kW (2 MMBtu/hr) | |
| Swirl mechanism | Adjustable air register; clockwise/counterclockwise operation | |
| | Primary air at burner | |
| | Secondary air around burner via straight throats | |
| Geometry | | |
| Throat diameter | 18 cm | (7 in.) |
| Divergent angle | 45° | |
| L/D | 1.0 | |
| Nominal residence time range | 3.5 to 8.7s | |
| Primary fuel | Propane | |
| Liquid feed system | Viking gear pump with recirculation loop: 32 ml/s (30 gph) at 420 kPa (61 psig) | |
| Temperature | 1,425°C (2,600°F) maximum operating | |
| **Characteristics of the Afterburner Chamber** | | |
| Length | 4.43m | (14.5 ft) |
| Inside diameter | 0.88m | (2.9 ft) |
| Outside diameter | 1.35m | (4.4 ft) |
| Chamber volume | 2.74 $m^3$ | (96 $ft^3$) |
| Shell construction | 0.63 cm | (0.25 in.) thick cold rolled steel |
| Refractory | | |
| Outside layer | 15.2 cm | (6 in.) thick Kaolite insulating refractory |
| Inside layer | 7.6 cm | (3 in.) thick Kaocrete 1,650°C (3,000°F) high alumina refractory |
| Burner | None | |

TABLE 1. DESIGN CHARACTERISTICS OF THE CRF LIQUID INJECTION INCINERATOR (CONCLUDED)

| | |
|---|---|
| **Characteristics of the Afterburner Chamber (concluded)** | |
| Nominal residence time | 3.5 to 8.7s |
| Temperature | 1,425°C (2,600°F) maximum operating |
| **Characteristics of the Transition Section** | |
| Length | 1.46m (4.8 ft) |
| Inside diameter | 0.88m (2.9 ft) |
| Outside diameter | 1.35m (4.4 ft) |
| Chamber volume | 0.89 $m^3$ (32 $ft^3$) |
| Refractory | Same as main and afterburner chambers |
| Total hot zone volume | 5.91 $m^3$ (209 $ft^3$) |
| **Characteristics of the Air Pollution Control Systems** | |
| System capacity | Inlet gas flow of 42 to 170 $m^3$/min (1,500 to 6,000 acfm) at 1,200°C (2,200°F), and 101 kPa (14.7 psig) |
| Pressure drop | Quench section: 0.5 to 075 kPa (2 to 3 in. WC)<br>Packed tower 1.0 kPa (4 in. WC) |
| Liquid flow | Quench section: 114 L/min (30 gpm)<br>Packed tower upper nozzle 95 L/min (25 gpm)<br>Packed tower lower nozzle 8.7 L/min (2.3 gpm)<br>Total scrubber system 218 L/min (57 gpm) |
| pH control | Feedback control by NaOH solution addition to scrubber liquid pump discharge |

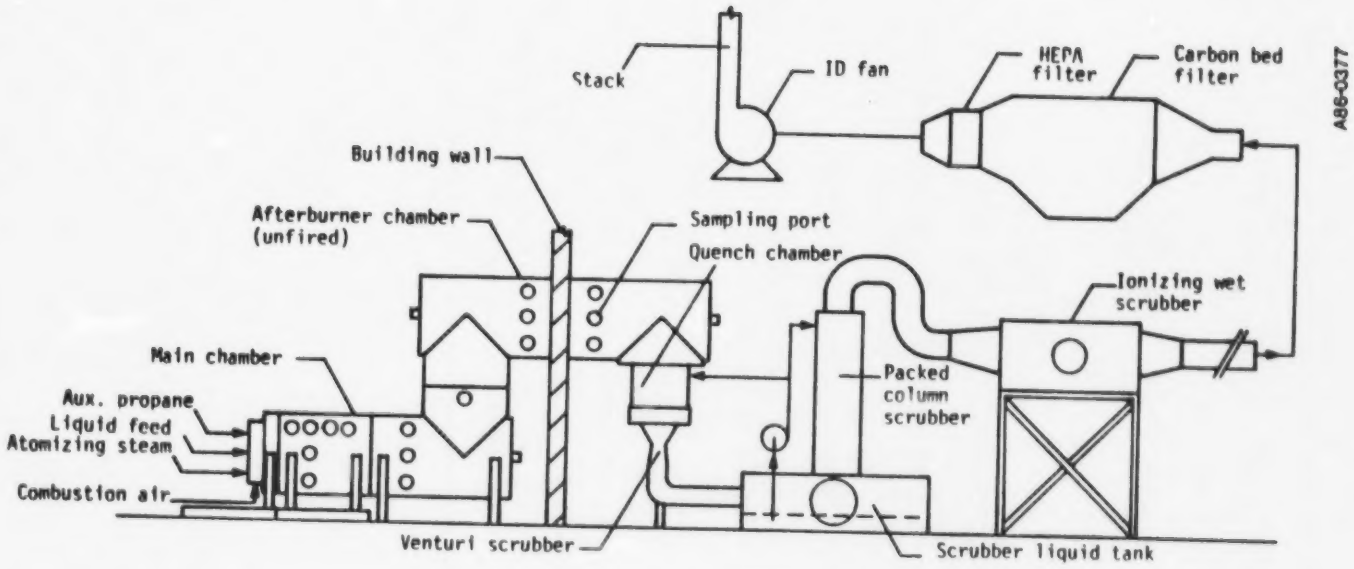



Figure 1. Simplified schematic of the EPA CRF liquid injection incinerator system.

pH sensor and controller monitors the liquor pH which was maintained above 6.0 to ensure proper HCl removal efficiency.

Downstream of the packed column scrubber, an ionizing-wet-scrubber (IWS) may be used to further remove particulate. During these tests, the IWS was not turned on.

As a redundant measure, downstream of the IWS an activated-carbon adsorber and a high efficiency particulate (HEPA) filter removed organic compounds and fine particulates, respectively. An induced-draft fan downstream of the HEPA filter draws a slight vacuum throughout the incinerator/scrubber system and vents the combustion products via a stack.

## 2.2 SYNTHETIC WASTE CHARACTERISTICS

As mentioned earlier, the feed liquids were synthetic mixtures of methanol, carbon tetrachloride, and chlorobenzene, spiked with antimony trichloride and arsenic trioxide. The mixtures were blended in various proportions to achieve the desired chlorine content. The originally-planned No. 2 fuel oil did not keep the antimony and arsenic compounds in solution and methanol was substituted for fuel oil.

Table 2 lists the targeted and the actual feed waste compositions. Two sets of actual compositions are reported in the table. One set was based on the constituent weights blended. The other set was based on analytical determination of the component concentrations in the composite feed samples collected during the tests. As noted in Section 1, only 12 of the target 18 test conditions were actually achieved. Data for the 6 conditions not achieved are therefore absent from Table 2.

The chlorine content in the feed material was one of the variables for this test series. By varying the concentrations of methanol (contains no Cl), chlorobenzene (contains 31.6 percent Cl) and carbon tetrachloride (contains 92.2 percent Cl), feed material with target chlorine contents of 0, 8, 18, 28, and 36 percent were prepared.

Antimony trichloride and arsenic trioxide were weighed and added to the methanol-based waste to about 40 ppm of antimony and 12 ppm of arsenic for each test. The trace element concentrations were planned to be identical for all tests. These concentrations in the feed material ensure that the worst-case concentrations in the stack gas will never exceed the threshold limit values (TLV, 0.2 mg As/$m^3$ and 0.5 mg Sb/$m^3$) for occupational exposure. Furthermore, if all the trace metals leave the incinerator via the scrubber blowdown water, their concentrations will not exceed the EP-toxicity limits (5 mg As/L, no limit established for Sb).

Table 2 indicated that the measured antimony and arsenic concentrations deviated significantly from the targeted values. Analyzed antimony concentrations ranged from 33 to 87 with a mean of 54 ppm, versus target prepared concentrations of about 40 ppm. Analyzed arsenic concentrations ranged from 1.9 to 14.5 with a mean of 5.4 ppm. Analyzed arsenic

# TABLE 2. TRACE ELEMENT TEST FEED COMPOSITION SUMMARY

| Test no. | Date | Calculated composition based on mixture proportions: Composition: Methanol (%) | Chloro-benzene (%) | Carbon tetra-chloride (%) | Sb (ppm) | As (ppm) | Ultimate analysis: C (%) | H (%) | O (%) | Cl (%) | Heating value: MJ/kg | Btu/lb | Measured composition based on analysis: Composition: Chloro-benzene (%) | Carbon tetra-chloride (%) | Sb (ppm) | As (ppm) | Ultimate analysis: C (%) | H (%) | O (%) | Cl (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1-21-87 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 2 | 1-27-87 | 65.3 | 23.2 | 11.5 | 40.2 | 12.1 | 40.2 | 9.2 | 32.7 | 17.9 | 21.5 | 9,232 | 22.7 | 12.2 | 52 | 2.8 | 38.5 | 9.1 | 33.6 | 18.8 |
| 3 | 2-18-87 | 64.9 | 24.1 | 11.0 | 40.0 | 12.1 | 40.6 | 9.2 | 32.5 | 17.7 | 21.6 | 9,300 | 27.3 | 15.1 | 45 | 3.1 | 38.5 | 9.1 | 33.6 | 18.8 |
| 4 | 1-29-87 | 54.2 | 23.9 | 21.8 | 40.0 | 12.1 | 37.4 | 7.8 | 27.1 | 27.7 | 19.3 | 8,281 | 24.9 | 27.9 | NA | NA | 36.1 | 8.1 | 29.4 | 26.4 |
| 5 | 2-05-87 | 55.0 | 23.8 | 21.3 | 38.8 | 11.8 | 37.5 | 7.9 | 27.5 | 27.1 | 19.4 | 8,331 | 25.3 | 33.2 | 24 | 3.2 | NA | NA | NA | NA |
| 6 | 2-19-87 | 75.8 | 24.1 | 0.0 | 40.0 | 12.1 | 43.9 | 10.6 | 37.9 | 7.6 | 24.0 | 10,324 | 26.1 | 0.0 | 33 | 3.0 | NA | NA | NA | NA |
| 7 | 1-30-87 | 54.0 | 23.9 | 22.2 | 40.0 | 12.1 | 37.2 | 7.8 | 27.0 | 28.0 | 19.2 | 8,250 | 25.9 | 28.0 | 64 | 14.5 | 36.1 | 8.1 | 29.4 | 26.4 |
| 8 | 2-05-87 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 9 | 2-20-87 | 76.0 | 24.0 | 0.0 | 40.1 | 12.1 | 43.8 | 10.6 | 38.0 | 7.6 | 24.0 | 10,322 | 26.3 | 0.0 | 33 | 9.4 | 44.7 | 10.5 | 36.9 | 7.9 |
| 10 | 2-03-87 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 11 | 2-05-87 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 12 | 2-18-87 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 13 | 2-10-87 | 65.0 | 24.0 | 11.0 | 40.0 | 12.1 | 40.6 | 9.2 | 32.5 | 17.7 | 21.6 | 9,294 | 26.3 | 12.7 | 87 | 8.9 | 38.5 | 9.1 | 33.6 | 18.8 |
| 14 | 2-24-87 | 44.9 | 25.6 | 29.5 | 40.0 | 12.1 | 35.5 | 6.7 | 22.5 | 35.3 | 17.7 | 7,607 | 26.8 | 31.4 | 60 | 2.5 | 36.3 | 6.9 | 23.0 | 33.8 |
| 15 | 2-26-87 | 100.0 | 0.0 | 0.0 | 40.0 | 12.1 | 37.5 | 12.5 | 50.0 | 0.0 | 22.7 | 9,765 | 0.0 | 0.0 | NA | NA | 35.6 | 12.2 | 52.2 | 0.0 |
| 16 | 2-12-87 | 65.0 | 24.0 | 11.0 | 40.0 | 12.1 | 40.6 | 9.2 | 32.5 | 17.7 | 21.6 | 9,294 | 27.8 | 12.8 | 70 | 3.8 | 38.5 | 9.1 | 33.6 | 18.8 |
| 17 | 2-18-87 | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 18 | 2-13-87 | 66.1 | 22.9 | 11.0 | 40.7 | 12.3 | 40.3 | 9.3 | 33.0 | 17.4 | 21.5 | 9,263 | 29.4 | 13.4 | 67 | 1.9 | 38.5 | 9.1 | 33.6 | 18.8 |

--: Test not performed; target conditions not achievable
NA: Analysis not performed

concentrations were generally substantially below the planned and blended concentration target of about 12 ppm. The reasons for the variability in feed concentrations and the low measured As concentration compared to target concentrations in the composite fuel samples are unknown. However, one could speculate that the variability in values may have been due to inhomogeneities in the feed mixture. In the tests, a measured amount of a $As_2O_3/SbCl_3$ mixture was poured into the feed liquid tank just prior to the start of liquid feed for a given test. The liquid feed tank was stirred. However, if the stirring were not complete, a homogeneous mixture would not have resulted. Thus, a composite feed sample obtained by taking a number of discrete aliquots from the liquid feed line to the burner might not have had the expected concentration corresponding to a homogeneous mixture.

The low As concentrations may have been due to an inability to dissolve all the $As_2O_3$ in the liquid feed. If undissolved $As_2O_3$ existed, then it may have been filtered out of the feed stream by the liquid feed strainer, may have settled to the bottom of the feed tank, or may have adhered to phase boundaries (liquid surface, tank walls, or the stirrer surfaces).

## 2.3 TEST CONDITIONS

The test program studied the effects of feed chlorine content and two incinerator operating parameters: incinerator temperature and combustion excess air. A Box-Wilson factorial experimental design (2) specified 18 test conditions for 5 levels of variation of each parameters.

Targeted chlorine contents were 0, 8, 18, 28 and 36 percent. Incinerator temperature targets were 1,045°, 1,130°, 1,250°, 1,365°, and 1,450°C (1,915°, 2,070°, 2,280°, 2,490°, and 2,640°F). Target incinerator exit flue gas oxygen concentrations were 5.5, 6.7, 8.4, 10.1, and 11.3 percent. Table 3 lists the planned and achieved conditions. Twelve of the 18 planned conditions were completed. It was not possible to produce stable flames for test conditions 8, 10, 11, 12, and 17 so they were abandoned. The achieved conditions for test 1 deviated excessively from target conditions, so its test data were discarded. Feed chlorine contents noted in Table 3 correspond to the measured values noted in Table 2.

The liquid feed entered the main combustion chamber via a steam-atomized nozzle at about 45 kg/hr (100 lb/hr). Auxiliary propane maintained the incinerator at temperatures which ranged from 1,137° to 1,450°C (2,079° to 2,642°F). Combustion air entered via swirl registers in the burner to produce from 5.9 to 11.7 percent $O_2$ at the incinerator exit.

Table 4 summarizes the operating conditions for the 12 tests performed. The table includes waste feedrates, auxiliary propane feedrates, flue gas $O_2$ levels, incinerator temperatures, and other pertinent information. Figure 2 graphically shows the test conditions. Data from target test 1 (shaded symbol) were discarded because of excessive deviation from the planned condition. The dashed symbols represent test conditions that were not achievable because of flame instability.

TABLE 3. SUMMARY OF TEST CONDITIONS

| Test no. | Feed Cl content, percent | | Primary combustor temperature, °C (°F) | | Afterburner exit $O_2$, percent | | Note |
|---|---|---|---|---|---|---|---|
| | Target | Actual | Target | Actual | Target | Actual | |
| 1 | 18 | -- | 1,250 (2,280) | -- -- | 8.4 | -- | |
| 2 | 18 | 18.8 | 1,250 (2,280) | 1,336 (2,437) | 8.4 | 8.8 | |
| 3 | 18 | 18.8 | 1,250 (2,280) | 1,228 (2,243) | 8.4 | 8.4 | |
| 4 | 28 | 26.4 | 1,365 (2,490) | 1,307 (2,384) | 10.1 | 10.6 | |
| 5 | 28 | 27.1[a] | 1,130 (2,070) | 1,137 (2,079) | 10.1 | 11.0 | |
| 6 | 8 | 7.6[a] | 1,365 (2,490) | 1,290 (2,354) | 10.1 | 10.1 | |
| 7 | 28 | 26.4 | 1,365 (2,490) | 1,399 (2,551) | 6.7 | 6.7 | |
| 8 | 8 | -- | 1,130 (2,070) | -- -- | 10.1 | -- | b |
| 9 | 8 | 7.9 | 1,365 (2,490) | 1,425 (2,597) | 6.7 | 5.9 | |
| 10 | 28 | -- | 1,130 (2,070) | -- -- | 6.7 | -- | b |
| 11 | 8 | -- | 1,130 (2,070) | -- -- | 6.7 | -- | b |
| 12 | 18 | -- | 1,250 (2,280) | -- -- | 5.5 | -- | b |
| 13 | 18 | 18.8 | 1,250 (2,280) | 1,203 (2,198) | 11.3 | 11.7 | |
| 14 | 36 | 33.8 | 1,250 (2,280) | 1,293 (2,359) | 8.4 | 9.2 | |
| 15 | 0 | 0 | 1,250 (2,280) | 1,265 (2,310) | 8.4 | 8.1 | |
| 16 | 18 | 18.8 | 1,450 (2,640) | 1,450 (2,642) | 8.4 | 7.5 | |
| 17 | 18 | -- | 1,045 (1,915) | -- -- | 8.4 | -- | b |
| 18 | 18 | 18.8 | 1,250 (2,280) | 1,176 (2,148) | 11.3 | 10.8 | |

[a]Cl content not measured, based on calculated value from mixture prepared.
[b]Unable to attain target condition due to unstable flame.

# TABLE 4. INCINERATOR OPERATING CONDITIONS DURING THE TRACE METAL TESTS (AVERAGE/RANGE)

| Test no. | Date | Cl (wt %) | Feed heating value, MJ/kg (Btu/lb) | Liquid feed-rate, kg/hr (lb/hr) | Feed heat input, kW (x10$^3$ Btu/hr) | Propane flowrate, scm/hr (scfh) | Propane heat input, kW (x10$^3$ Btu/hr) | Total heat input, kW (x10$^3$ Btu/hr) | Combustion air flowrate, scm/min (scfm) | Afterburner exit flue gas $O_2$, (%) | Main chamber exit temperature, °C (°F) | Based on blend | | Based on analysis | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | Antimony feedrate, (g/hr) | Arsenic feedrate, (g/hr) | Antimony feedrate, (g/hr) | Arsenic feedrate, (g/hr) |
| 2 | 1-27-87 | 18.8 | 21.5 (9,232) | 41<br>20-50<br>(90)<br>(45-110) | 242<br>122-298<br>(827)<br>(415-1,016) | 11.2<br>9.6-12.2<br>(395)<br>(339-430) | 284<br>243-309<br>(968)<br>(831-1,054) | 526<br>365-606<br>(1,795)<br>(1,246-2,069) | 12.2<br>12.0-12.7<br>(431)<br>(425-447) | 9.2<br>7.5-11.5 | 1,336<br>1,267-1,373<br>(2,437)<br>(2,313-2,503) | 1.63 | 0.49 | 2.13 | 0.11 |
| 3 | 2-18-87 | 18.8 | 21.6 (9,300) | 45<br>33-53<br>(100)<br>(72-116) | 271<br>196-316<br>(926)<br>(670-1,079) | 5.3<br>4.6-5.9<br>(186)<br>(162-210) | 134<br>116-151<br>(456)<br>(397-515) | 405<br>312-467<br>(1,381)<br>(1,067-1,593) | 7.7<br>7.2-8.1<br>(271)<br>(256-285) | 8.6<br>7.5-9.5 | 1,228<br>1,056-1,293<br>(2,243)<br>(1,932-2,359) | 1.81 | 0.55 | 2.03 | 0.14 |
| 4 | 1-29-87 | 26.4 | 19.3 (8,281) | 41<br>36-58<br>(91)<br>(80-128) | 220<br>194-311<br>(751)<br>(662-1,060) | 11.0<br>11.0-11.3<br>(390)<br>(388-398) | 280<br>279-286<br>(956)<br>(951-975) | 500<br>473-596<br>(1,707)<br>(1,613-2,035) | 10.6<br>10.4-11.1<br>(374)<br>(368-391) | 10.5<br>9.5-16.5 | 1,307<br>1,243-1,361<br>(2,384)<br>(2,269-2,481) | 1.65 | 0.58 | -- | -- |
| 5 | 2-05-87 | 27.1 | 19.0 (8,332) | 42<br>17-58<br>(93)<br>(38-128) | 228<br>93-312<br>(778)<br>(317-1,066) | 5.8<br>4.7-6.1<br>(206)<br>(167-216) | 148<br>120-155<br>(505)<br>(409-529) | 376<br>213-468<br>(1,283)<br>(726-1,596) | 6.6<br>6.5-6.6<br>(232)<br>(231-233) | 10.8<br>7.0-14.0 | 1,137<br>1,040-1,248<br>(2,079)<br>(1,904-2,278) | 1.69 | 0.51 | 1.26 | 0.17 |
| 6 | 2-19-87 | 7.6 | 24.0 (10,324) | 46<br>39-52<br>(101)<br>(87-114) | 304<br>263-345<br>(1,038)<br>(898-1,177) | 5.5<br>5.3-5.9<br>(195)<br>(188-210) | 140<br>135-151<br>(478)<br>(461-515) | 444<br>398-496<br>(1,516)<br>(1,359-1,691) | 10.6<br>9.7-9.9<br>(375)<br>(341-351) | 9.8<br>9.0-11.5 | 1,290<br>1,241-1,394<br>(2,354)<br>(2,265-2,541) | 1.82 | 0.55 | 1.52 | 0.14 |
| 7 | 1-30-87 | 26.4 | 19.2 (8,250) | 43<br>22-51<br>(95)<br>(48-112) | 229<br>116-271<br>(781)<br>(396-924) | 7.9<br>7.7-8.2<br>(278)<br>(273-288) | 200<br>196-207<br>(681)<br>(669-706) | 428<br>312-477<br>(1,462)<br>(1,065-1,628) | 6.3<br>6.2-7.3<br>(224)<br>(220-258) | 6.1<br>5.5-8.0 | 1,398<br>1,276-1,448<br>(2,551)<br>(2,328-2,638) | 1.72 | 0.52 | 2.75 | 0.62 |
| 9 | 2-20-87 | 7.9 | 24.0 (10,320) | 46<br>33-59<br>(102)<br>(72-130) | 309<br>218-393<br>(1,055)<br>(743-1,342) | 7.2<br>4.6-8.2<br>(256)<br>(162-290) | 184<br>116-208<br>(627)<br>(397-711) | 493<br>334-601<br>(1,683)<br>(1,140-2,052) | 8.2<br>6.6-8.2<br>(291)<br>(233-291) | 5.8<br>3.0-9.0 | 1,425<br>1,244-1,495<br>(2,597)<br>(2,272-2,723) | 1.86 | 0.56 | 1.52 | 0.43 |
| 13 | 2-10-87 | 18.8 | 21.6 (9,294) | 47<br>43-53<br>(103)<br>(94-116) | 281<br>256-316<br>(959)<br>(874-1,078) | 5.8<br>5.4-6.0<br>(206)<br>(191-213) | 148<br>137-153<br>(505)<br>(468-522) | 429<br>393-469<br>(1,464)<br>(1,342-1,600) | 8.4<br>8.2-8.8<br>(296)<br>(289-310) | 11.8<br>11.3-12.5 | 1,203<br>1,162-1,286<br>(2,198)<br>(2,123-2,346) | 1.87 | 0.57 | 4.09 | 0.42 |

## TABLE 4. INCINERATOR OPERATING CONDITIONS DURING THE TRACE METAL TESTS (AVERAGE/RANGE) (CONCLUDED)

| Test no. | Date | Cl (wt %) | Feed heating value, MJ/kg (Btu/lb) | Liquid feed-rate, kg/hr (lb/hr) | Feed heat input, kW ($\times 10^3$ Btu/hr) | Propane flowrate, scm/hr (scfh) | Propane heat input, kW ($\times 10^3$ Btu/hr) | Total heat input, kW ($\times 10^3$ Btu/hr) | Combustion air flowrate, scm/min (scfm) | Afterburner exit flue gas $O_2$, (%) | Main chamber exit temperature, °C (°F) | Based on blend | | Based on analysis | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | Antimony feedrate, (g/hr) | Arsenic feedrate, (g/hr) | Antimony feedrate, (g/hr) | Arsenic feedrate, (g/hr) |
| 14 | 2-24-87 | 33.8 | 17.7 (7,607) | 49 37-58 (108) (82-128) | 241 183-285 (822) (624-974) | 5.9 5.9-5.9 (210) (210-210) | 151 151-151 (515) (515-515) | 392 334-436 (1,336) (1,138-1,488) | 7.2 6.8-8.7 (256) (240-306) | 9.0 6.0-14.0 | 1,293 1,252-1,359 (2,359) (2,286-2,479) | 1.96 | 0.59 | 2.94 | 0.12 |
| 15 | 2-26-87 | 0.0 | 22.7 (9,765) | 44 35-49 (96) (78-108) | 275 223-309 (940) (762-1,055) | 4.7 4.6-5.3 (165) (161-186) | 118 116-134 (404) (394-456) | 394 339-443 (1,345) (1,156-1,510) | 7.2 7.1-7.2 (253) (252-254) | 7.7 6.0-9.0 | 1,266 1,173-1,312 (2,310) (2,144-2,394) | 1.75 | 0.53 | 2.64 | 0.11 |
| 16 | 2-12-87 | 18.8 | 21.6 (9,294) | 45 40-61 (99) (88-134) | 268 240-365 (915) (818-1,245) | 5.9 5.3-5.9 (208) (188-210) | 149 135-151 (510) (461-515) | 418 375-516 (1,425) (1,278-1,760) | 7.7 7.6-8.1 (272) (270-286) | 7.4 2.8-10.5 | 1,450 1,398-1,486 (2,642) (2,549-2,707) | 1.79 | 0.54 | 3.15 | 0.17 |
| 18 | 2-13-87 | 18.8 | 21.5 (9,263) | 45 39-59 (99) (86-129) | 268 233-350 (916) (797-1,195) | 5.9 5.9-5.9 (210) (210-210) | 151 151-151 (515) (515-515) | 419 384-501 (1,431) (1,311-1,709) | 11.2 10.3-12.0 (396) (365-424) | 10.7 8.0-11.0 | 1,176 1,161-1,210 (2,148) (2,122-2,210) | 1.83 | 0.55 | 3.02 | 0.09 |

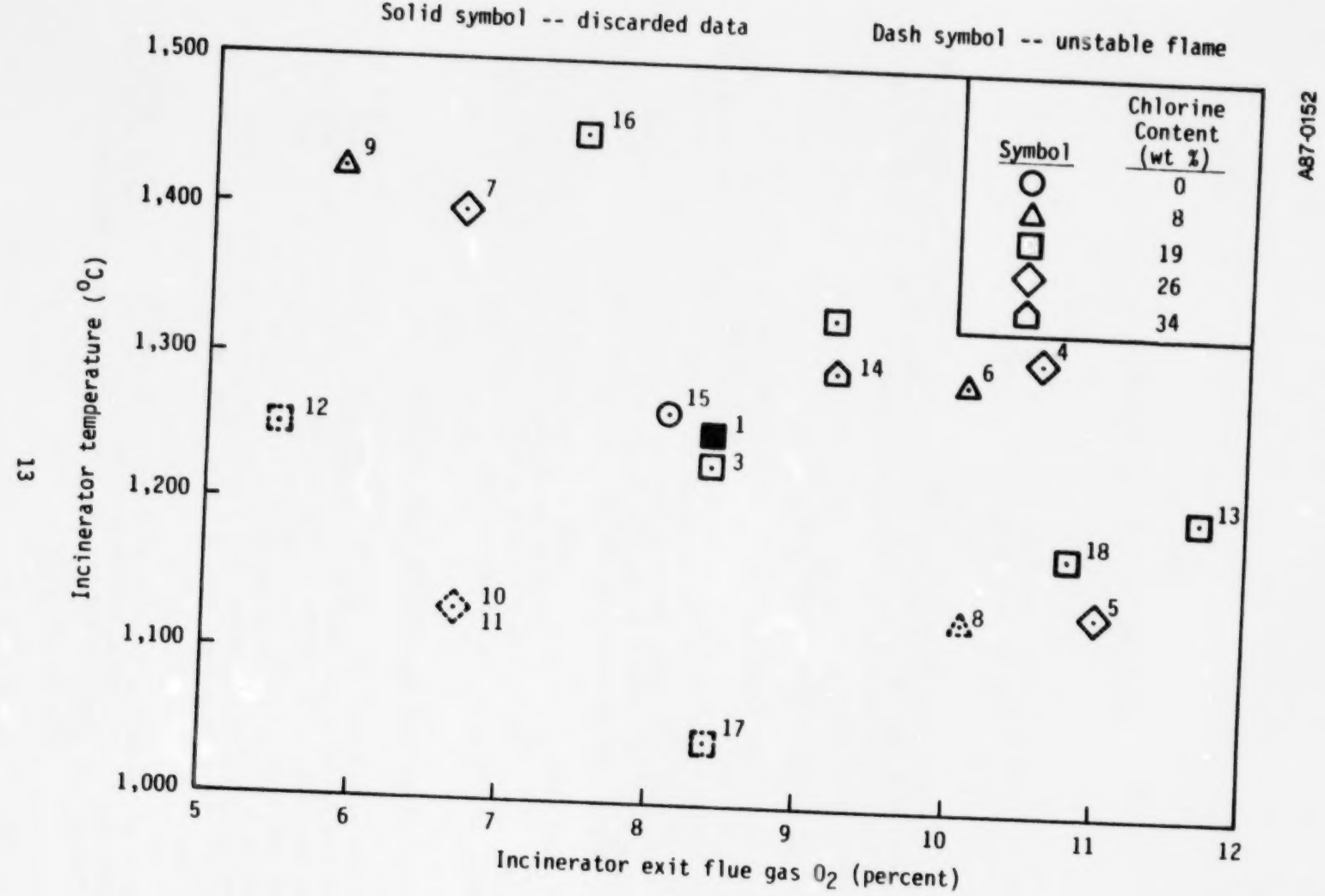



Figure 2. Trace element test conditions.

## SECTION 3

## SAMPLING AND ANALYSIS PROTOCOL

In order to achieve the objectives of the test program, an extensive sampling and analysis program was performed. This section summarizes the protocols and methods used.

Figure 3 summarizes the sampling effort. Table 5 lists the sampling and analysis protocols used which included continuous $O_2$, CO, and $CO_2$ monitors, volatile organic sampling trains, and Method 5 sampling trains modified for trace element sampling.

### 3.1 VOLATILE ORGANIC COMPOUND SAMPLING

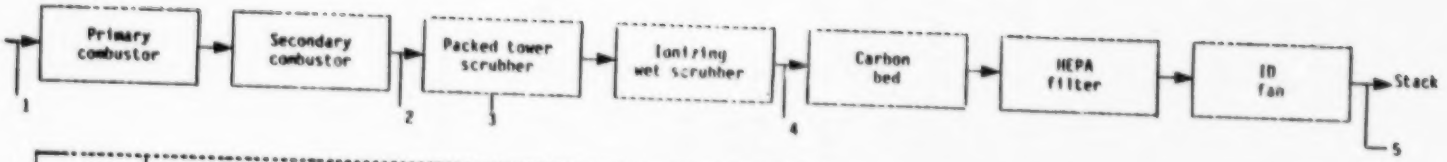
Volatile organic sampling trains (VOST) were used to collect samples at the afterburner and the scrubber exits. Three trap pairs were taken for each test. Each trap pair typically required 20 minutes and withdrew 20 liters of sample gas through the resin. Immediately following the sample collection, the CRF Analytical Chemistry Staff performed the analysis for 22 volatile organic compounds by thermal desorption purge and trap, flame ionzation detector (FID). The 22 compounds analyzed for are listed in Table 6.

Original test plans were to conduct simultaneous sampling at the afterburner exit and at the scrubber discharge. However, Teflon probe lining failure for one of the CRF VOST trains rendered it unavailable for most of the tests. Consequently, simultaneous sampling was only successful for one test.

### 3.2 TRACE ELEMENT AND PARTICULATE LOAD SAMPLING

At the afterburner exit and the scrubber exit, EPA Reference Method 5 sampling trains collected samples for particulate load and arsenic and antimony analyses. The Method 5 train was modified to enhance collection of antimony and arsenic. The train consisted of a probe and a glass cyclone, followed by a filter and five impingers. As noted in Table 7, the first impinger contained 0.1 N NaOH. The second, third and fourth impingers contained 0.2 M $(NH_4)_2S_2O_8$ + 0.02 M $AgNO_3$. The last impinger contained silica gel.

Each of the Method 5 trains produced seven samples for element analyses: one probe and cyclone wash, one particulate filter, and 5 impinger solutions. The probe and cyclone wash and the impinger solutions were adjusted with




| Sampling point | Parameter | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Waste feedrate | Waste feed | Auxiliary fuel feedrate | Air feedrate | Scrubber blowdown | CM's ($O_2$, CO, $CO_2$, $NO_x$) | Volume flow | MS (particulate, metals) | VOST (volatile organics) | Temperature | Pressure | Relative humidity |
| 1 | X | X | X | X | | | | | | | | |
| 2 | | | | | | X | X | X | X | X | X | X |
| 3 | | | | | X | | | | | | | |
| 4 | | | | | | | X | | | | | |
| 5 | | | | | | | X | X | X | X | X | X |
| | | | | | | X | X | X[a] | | X | X | X |

[a]Particulate only.

Figure 3. Sampling Protocol

TABLE 5. SAMPLING AND ANALYSIS MATRIX SUMMARY

| Sample | Location | Sampling procedure | Analysis | | |
|---|---|---|---|---|---|
| | | | Parameter | Method | Frequency |
| Feed material | Primary combustor inlet | Composite sample | $CCl_4$, $C_6H_5Cl$ | Dilution; GC/MS by Method 8270[a] | All tests |
| | | | Ultimate analysis (C,H,O,Cl) | A003[b] | All tests |
| | | | As | Digestion by Method 3020, AA by Method 7060[a] | All tests |
| | | | Sb | Digestion by Method 3020, analysis by Method 7041[a] | All Tests |
| Scrubber blowdown water | Blowdown discharge | Composite sample | As | Digestion by Method 3020, AA by Method 7060[a] | All tests |
| | | | Sb | Digestion by Method 3020, analysis by Method 7041[a] | All tests |
| Flue gas particulate and trace elements | Secondary combustion chamber exit and ionizing wet scrubber exit | Method 5[c] | Particulate load | Method 5[c] | All tests |
| | | | As | Digestion by Method 3020, AA by Method 7060[a] | All tests |
| | | | Sb | Digestion by Method 3060, analysis by Method 7041[a] | All tests |
| Flue gas volatile organics | Secondary combustion chamber exit and the ionizing wet scrubber exit | Volatile organic sampling train (VOST)[d] | 22 FID volatile organics | Thermal desorption, purge and trap GC/FID in accordance with Method 8010[a] | All tests |
| Stack gas particulate emissions | Stack downstream of carbon bed/ HEPA filter | Method 5[c] | Particulate load | Method 5[c] | All tests |

[a]Reference 3
[b]Reference 4
[c]Reference 5
[d]Reference 6

TABLE 6. VOLATILE ORGANIC COMPOUNDS ROUTINELY ANALYZED BY GC/FID AT THE CRF

| | |
|---|---|
| Methylene chloride | Trichloroethylene |
| 1,1-Dichloroethylene | Benzene |
| 1,1-Dichloroethane | 1,1,2-Trichloroethane |
| t-1,2-Dichloroethylene | Hexane |
| Chloroform | Bromoform |
| 1,2-Dichloroethane | Tetrachloroethylene + Tetrachloroethane |
| 1,1,1-Trichloroethane | Toluene + Heptane |
| Carbon Tetrachloride | Chlorobenzene |
| Bromodichloromethane | 1,3-Dichlorobenzene |
| 1,2-Dichloropropane | 1,2-Dichlorobenzene |
| t-1,3-Dichloropropylene | 1,4-Dichlorobenzene |

TABLE 7. METHOD 5 TRAIN IMPINGER SYSTEM REAGENTS

| Impinger No. | Reagents | Quantity | Purpose |
|---|---|---|---|
| 1 | 0.1 N NaOH | 500 mL | Trap acidic HCl reducing gases |
| 2-4 | 0.2 M $(NH_4)_2S_2O_8$ +0.02 M $AgNO_3$ | 500 mL | Collection of volatile trace elements by oxidative dissolution |
| 5 | Silica gel | 750 g | Prevent moisture from reaching pumps |

nitric acid to a pH of less than 2. The filter samples were suspended in about 100 ml of deionized water and similarly adjusted to pH less than 2. The samples were then sent to an offsite laboratory for digestion; and arsenic and antimony analysis. Following sample preparation, atomic absorption spectrometry employing a stabilized-temperature graphite-platform furnace was used to analyze for antimony and arsenic concentrations.

In order to minimize errors that might be introduced during sample preparation steps, it was decided that the probe wash samples would not be evaporated and weighed before antimony and arsenic analyses. Consequently, the particulate contributions from the probe wash samples collected at the afterburner exit and the scrubber exit could not be determined. Only the filter particulate samples were dessicated to constant weight to determine the particulate weights which defined the particulate loading at the afterburner and scrubber exits.

# SECTION 4

# TEST RESULTS

Table 3 noted that 12 out of the 18 target tests were completed. The remaining six tests were generally unattainable due to flame stability problems at low flame temperatures and low incinerator excess air levels. The following subsection will discuss the data from the 12 completed tests.

## 4.1 TRACE ELEMENT DISCHARGES

### 4.1.1 Antimony Discharge Distributions

#### Distribution Between Effluent Streams

Discharge distributions for antimony in the following paragraphs are discussed in terms of the flowrate of the antimony measured in the afterburner exit flue gas, the scrubber exit flue gas, and scrubber blowdown discharge expressed as a fraction of the inlet antimony feedrate. Two methods were used to determine the antimony feed rates. One was based on the analyzed antimony concentrations in composite feed samples, the other was based on antimony concentrations as determined by the feed mixture blending proportions. The following discussion is based on the measured antimony feed concentrations. In any case, observations would not alter substantially if the mixture preparation data were used.

Table 8 summarizes the fractions of the antimony found in the afterburner exit flue gas, the scrubber exit flue gas, and the scrubber blowdown water. As shown in the table, the three tests where the feed contained nominally 26 percent chlorine, no more than about 1 percent of the inlet antimony was measured in the afterburner exit flue gas. For one test with the nominally 19 percent chlorine feed, less than 0.1 percent of the inlet antimony was detected in the afterburner exit flue gas. For the remaining tests with 19 percent chlorine feed and the tests with nominally 8 percent and 0 percent chlorine feed, between 25 and 101 percent of the input antimony was measured in the afterburner exit flue gas.

Between 15 and 84 percent of the inlet antimony was measured in scrubber discharge streams (flue gas plus blowdown). Scrubber blowdown water accounted for 5 to 75 percent of the inlet antimony. Scrubber exit flue gas accounted for between 3 and 21 percent of the inlet antimony. Comparable fractions of antimony were generally measured in these two streams for a given test.

TABLE 8. ANTIMONY DISCHARGE DISTRIBUTIONS

| Test No. | Feed Cl content, percent | Primary combustor temperature, °C (°F) | Afterburner exit $O_2$, percent | Antimony discharge distribution[a] (percent of feedrate) | | | |
|---|---|---|---|---|---|---|---|
| | | | | Afterburner exit flue gas | Scrubber Discharge Streams | | |
| | | | | | Flue gas | Blowdown | Total |
| 14 | 33.8 | 1,293 (2,359) | 9.2 | 34 (52) | 8 (12) | 7 (11) | 15 (23) |
| 4 | 26.4 | 1,307 (2,384) | 10.5 | 0.18[b] | 14[b] | 22[b] | 36[b] |
| 5 | 27.1 | 1,137 (2,079) | 10.8 | 0.3 (0.24) | 17 (13) | 21 (16) | 38 (29) |
| 7 | 26.4 | 1,399 (2,551) | 6.1 | 1.1 (1.8) | 18 (30) | 8 (12) | 26 (42) |
| 13 | 18.8 | 1,203 (2,198) | 11.8 | 0 (0.11) | 3 (6.6) | 40 (87) | 43 (94) |
| 18 | 18.8 | 1,176 (2,148) | 10.7 | 32 (53) | 9 (14) | 75 (124) | 84 (138) |
| 16 | 18.8 | 1,450 (2,642) | 7.4 | 83 (146) | 21 (36) | 5 (8.3) | 26 (44) |
| 2 | 18.8 | 1,336 (2,437) | 9.2 | 79 (104) | 15 (20) | 9 (12) | 24 (32) |
| 3 | 18.8 | 1,228 (2,243) | 8.4 | 89 (99) | 10 (11) | 13 (15) | 23 (26) |
| 6 | 7.6 | 1,290 (2,354) | 10.1 | 56 (47) | 20 (17) | 10 (18) | 30 (35) |
| 9 | 7.9 | 1,425 (2,597) | 5.9 | 101 (83) | 17 (14) | 18 (15) | 35 (29) |
| 15 | 0 | 1,265 (2,310) | 8.1 | 25 (38) | 9.4 (14) | 7 (11) | 16 (25) |

[a]Numbers in parenthesis are based on a feed composition calculated from feed constituent blending proportions

[b]No feed sample analysis; value based on a feed composition calculated from feed constituent blending proportions

Mass balance closure results for antimony were mixed. Mass balance closure in the afterburner exit flue gas was quite poor for four tests as noted above. However, for the remaining tests (4 of 5 at the nominally 19 percent chlorine feed, two at nominally 8 percent chlorine feed, the one with no, and the one with about 34 percent chlorine feed) mass balance closure is better. Past attempts to close a trace element mass balance around a combustion process have generally been unsuccessful. Closure within a factor of 3 has generally been the best achievable. Eight tests met this level of closure.

Closure results are generally better at the scrubber discharge. Still, closure within a factor of 3 was obtained for only 7 tests.

The inability to attain better mass balance closure in these tests was likely affected by the fact that accumulation of element concentrations in the recirculating scrubber liquor was not taken into account.

Distribution Between Vapor and Solid in Flue Gas

Table 9 shows the distribution of antimony between the particulate and vapor phases in the flue gas at both the afterburner exit and the scrubber exit. The table shows that, at the afterburner exit where temperatures were above 760°C (1,400°F), the vapor phase (impinger samples) contained an average of 55 percent (range 5 to 90) of the collected antimony. At the scrubber exit where the temperature was generally at 74°C (165°F), the vapor phase accounted for an average of 6 percent (range 0 to 38) of the collected antimony.

### 4.1.2 Arsenic Discharge Distributions

Distribution between Effluent Streams

Table 10 presents a summary of the fractions of arsenic measured in the afterburner exit flue gas and in the scrubber discharge stream (flue gas and blowdown) analogous to the Table 8 summary for antimony. In the afterburner exit flue gas, detectable levels of arsenic were measured for 9 tests. The measured fractions of feed arsenic ranged from 1.4 to 245 percent. Eight tests showed better than 10 percent arsenic recovery. Seven of these corresponded to mass balance closure within a factor of 3.

The combination of the scrubber exit gas with the scrubber blowdown accounted for 3.8 to 299 percent of the input arsenic. Mass balance closure around the scrubber was within a factor of 3 for eight tests. More often than not, a greater proportion of arsenic was found in the flue gas than in the scrubber blowdown.

Distribution Between Vapor and Solid in Flue Gas

Table 11 summarizes the arsenic phase distribution data for the flue gas at the two locations sampled. At the afterburner exit, arsenic is distributed between the vapor and particulate phases. More often than not a

TABLE 9. ANTIMONY FLUE GAS DISTRIBUTIONS

| Test No. | Feed Cl content, percent | Primary combustor temperature, °C (°F) | Afterburner exit $O_2$, percent | Antimony distribution between particulate and vapor phase in the flue gas (percent) | | | |
|---|---|---|---|---|---|---|---|
| | | | | Afterburner Exit | | Scrubber Discharge | |
| | | | | Particulate | Vapor | Particulate | Vapor |
| 14 | 33.8 | 1,293 (2,359) | 9.2 | 32 | 68 | 99 | 1 |
| 4 | 26.4 | 1,307 (2,384) | 10.5 | 62 | 38 | 99 | 1 |
| 5 | 27.1 | 1,137 (2,079) | 10.8 | 49 | 51 | 62 | 38 |
| 7 | 26.4 | 1,399 (2,551) | 6.1 | 53 | 47 | 99 | 1 |
| 13 | 18.8 | 1,203 (2,198) | 11.8 | 42 | 58 | 99 | 1 |
| 18 | 18.8 | 1,176 (2,148) | 10.7 | 21 | 79 | 100 | 0 |
| 16 | 18.8 | 1,450 (2,642) | 7.4 | 76 | 24 | 99.9 | 0.1 |
| 2 | 18.8 | 1,336 (2,437) | 9.2 | 10 | 90 | 82 | 18 |
| 3 | 18.8 | 1,228 (2,243) | 8.4 | 22 | 78 | 97 | 3 |
| 6 | 7.6 | 1,290 (2,354) | 10.1 | 41 | 59 | 100 | 0 |
| 9 | 7.9 | 1,425 (2,597) | 5.9 | 34 | 66 | 98 | 2 |
| 15 | 0 | 1,265 (2,310) | 8.1 | 95 | 5 | 99 | 1 |

TABLE 10. ARSENIC DISCHARGE DISTRIBUTIONS

| Test No. | Feed Cl content, percent | Primary combustor temperature, °C (°F) | Afterburner exit $O_2$, percent | Arsenic discharge distribution[a] (percent of feedrate) | | | |
|---|---|---|---|---|---|---|---|
| | | | | Afterburner exit flue gas | Scrubber Discharge Streams | | |
| | | | | | Flue gas | Blowdown | Total |
| 14 | 33.8 | 1,293 (2,359) | 9.2 | 126 (26) | 30 (6.1) | 2.5 (0.5) | 33 (6.6) |
| 4 | 26.4 | 1,307 (2,384) | 10.5 | ND | 9.0[b] | 3.8[b] | 12[b] |
| 5 | 27.1 | 1,137 (2,079) | 10.8 | ND | 15 (5.1) | 6.9 (2.3) | 22 (7.4) |
| 7 | 26.4 | 1,399 (2,551) | 6.1 | 1.4 (1.7) | 49 (58) | 5.4 (6.4) | 54 (64) |
| 13 | 18.8 | 1,203 (2,198) | 11.8 | ND | ND | 3.8 (2.8) | 3.8 (2.8) |
| 18 | 18.8 | 1,176 (2,148) | 10.7 | 25 (4.0) | 13 (2.2) | 19 (3.1) | 32 (5.3) |
| 16 | 18.8 | 1,450 (2,642) | 7.4 | 245 (77) | 30 (9.4) | 6.0 (1.9) | 36 (11) |
| 2 | 18.8 | 1,336 (2,437) | 9.2 | 45 (10.2) | 19 (4.3) | 2.3 (0.51) | 21 (4.8) |
| 3 | 18.8 | 1,228 (2,243) | 8.4 | 77 (20) | 289 (73) | 9.8 (2.5) | 299 (76) |
| 6 | 7.6 | 1,290 (2,354) | 10.1 | 125 (32) | 60 (15) | 21 (5.3) | 81 (20) |
| 9 | 7.9 | 1,425 (2,597) | 5.9 | 153 (177) | 26 (20) | 20 (15) | 46 (35) |
| 15 | 0 | 1,265 (2,310) | 8.1 | 103 (21) | 5 (1.1) | 32 (6.6) | 37 (7.7) |

ND -- Not detected.

[a]Numbers in parenthesis are based on a feed composition calculated from feed constituent blending proportion

[b]Feed sample not analyzed; value based on feed composition calculated from feed constituent blending proportion

TABLE 11. ARSENIC FLUE GAS DISTRIBUTIONS

| Test No. | Feed Cl content, percent | Primary combustor temperature, °C (F°) | Afterburner exit $O_2$, percent | Arsenic distribution between particulate and vapor phase in the flue gas (percent) | | | |
|---|---|---|---|---|---|---|---|
| | | | | Afterburner exit | | Scrubber discharge | |
| | | | | Particulate | Vapor | Particulate | Vapor |
| 14 | 33.8 | 1,293 (2,359) | 9.2 | 70 | 30 | 85 | 15 |
| 4 | 26.4 | 1,307 (2,384) | 10.5 | ND | ND | 100 | 0 |
| 5 | 27.1 | 1,137 (2,079) | 10.8 | ND | ND | 83 | 17 |
| 7 | 26.4 | 1,399 (2,551) | 6.1 | 43 | 57 | 100 | 0 |
| 13 | 18.8 | 1,203 (2,198) | 11.8 | ND | ND | ND | ND |
| 18 | 18.8 | 1,176 (2,148) | 10.7 | 32 | 68 | 100 | 0 |
| 16 | 18.8 | 1,450 (2,642) | 7.4 | 76 | 24 | 100 | 0 |
| 2 | 18.8 | 1,336 (2,437) | 9.2 | 83 | 17 | 94 | 6 |
| 3 | 18.8 | 1,228 (2,243) | 8.4 | 41 | 59 | 100 | 0 |
| 6 | 7.6 | 1,290 (2,354) | 10.1 | 71 | 29 | 100 | 0 |
| 9 | 7.9 | 1,425 (2,597) | 5.9 | 69 | 31 | 100 | 0 |
| 15 | 0 | 1,265 (2,310) | 8.1 | 98 | 2 | 100 | 0 |

ND -- Not Detected.

greater proportion was found in the particulate. At the scrubber discharge most (usually all) of the arsenic was measured in the particulate.

### 4.1.3 Trace Element Discharge Summary

A statistical treatment of the data obtained in these tests to identify any correlations between antimony or arsenic discharge rates or discharge distributions and the primary test variables was planned. However, this analysis became complicated for two reasons. First, only 12 of the 18 tests in the factorial matrix were achieved in the test program. Second, deviations between actual test conditions and target conditions were substantial for many tests, especially for temperature and flue gas $O_2$. As a consequence, the mathematics involved in appropriately analyzing the data statistically became significantly more complicated. The result has been that, although such an analysis is still planned, results from this analysis are not available for reporting at present.

The major reason actual test conditions deviated from target conditions is that liquid feed, auxiliary fuel feed, and combustion air flowrates to the LIS could only be set and controlled manually. As a result, at the beginning of a test these were placed at settings expected to give target conditions, and adjusted periodically in an attempt to maintain target conditions. However, since the lag time between setting adjustment and pseudo-steady operation in the system is relatively long, it was difficult to achieve steady operation, and more so steady operation at target conditions, within the confines of a test day. Plans are to install automatic flow controllers for liquid feed, auxiliary fuel feed, and combustion air flowrates, and control these via a feedback control system. When this upgrade is complete, the ability to easily establish and maintain a given target operating condition will become possible.

Inspection of the data in Tables 8 through 11 shows no clear variation in trace element discharge results with the primary test variables. At present, only this conclusion will be stated.

## 4.2 PRINCIPAL ORGANIC HAZARDOUS CONSTITUENT DESTRUCTION AND REMOVAL EFFICIENCIES

If the test liquids fed in these tests were hazardous wastes, their principal organic hazardous constituents (POHCs) would be carbon tetrachloride and chlorobenzene. Feed liquid carbon tetrachloride concentrations ranged from 0 to 33.2 percent. Feed liquid chlorobenzene concentrations ranged from 0 to 29.4 percent. Table 12 summarizes the destruction and removal efficiency (DRE) data for these constituents based on the analyses of the VOST samples taken. As noted in Section 3, VOST sampling was performed simultaneously at the afterburner exit and the scrubber discharge for only one test. Only data for 10 tests are shown in Table 12. No VOST sampling was performed for test numbers 7 and 15. The liquid feed for test number 15 contained no chlorobenzene nor carbon tetrachloride so VOST testing was not planned. Sampling train problems prevented VOST testing for test number 7.

TABLE 12. POHC DREs

| Test No. | Afterburner exit $O_2$, percent | Combustor temperature, °C (°F) | Carbon Tetrachloride | | | Chlorobenzene | | |
|---|---|---|---|---|---|---|---|---|
| | | | Feed concentration, percent | DRE (percent) | | Feed concentration, percent | DRE (percent) | |
| | | | | Afterburner exit | Scrubber discharge | | Afterburner exit | Scrubber discharge |
| 14 | 9.2 | 1,293 (2,359) | 31.4 | --[a] | 99.99980 | 26.8 | --[a] | 99.9985 |
| 4 | 10.5 | 1,307 (2,384) | 27.9 | 99.9988 | --[a] | 24.9 | 99.9996 | --[a] |
| 5 | 10.8 | 1,137 (2,079) | 33.2 | --[a] | 99.999945 | 25.3 | --[a] | 99.999984 |
| 13 | 11.8 | 1,203 (2,198) | 12.7 | --[a] | 99.99983 | 26.3 | --[a] | 99.999987 |
| 18 | 10.7 | 1,176 (2,148) | 13.4 | --[a] | 99.99973 | 29.4 | --[a] | 99.999981 |
| 16 | 7.4 | 1,450 (2,642) | 12.8 | --[a] | 99.99953 | 27.8 | --[a] | 99.999981 |
| 2 | 9.2 | 1,336 (2,437) | 12.2 | 99.99982 | 99.99985 | 22.7 | 99.9995 | 99.999958 |
| 3 | 8.4 | 1,228 (2,243) | 15.1 | --[a] | 99.99987 | 27.3 | --[a] | 99.999946 |
| 6 | 10.1 | 1,290 (2,354) | 0 | --[b] | --[b] | 26.1 | --[a] | 99.999983 |
| 9 | 5.9 | 1,425 (2,597) | 0 | --[b] | --[b] | 26.3 | --[a] | 99.99985 |

[a]Data not available.
[b]This POHC not in feed.

As shown in Table 12, high DREs were attained for both carbon tetrachloride and chlorobenzene in all tests. Chlorobenzene DREs were generally higher than those for carbon tetrachloride. Carbon tetrachloride DREs were always greater than 99.99 percent at the afterburner exit. These increased to greater than 99.999 percent at the scrubber exit. Similarly, chlorobenzene DREs ranged from 99.999 percent or greater at the afterburner exit to generally greater than 99.9999 percent at the scrubber exit (one test, test 14, had lower chlorobenzene DRE at the scrubber exit). The available data do not suggest any clear effect of temperature, feed composition nor excess air level on DRE. This is in general agreement with previous CRF data which have consistently shown high DREs for POHCs present in feed mixtures at relatively high concentration.

## 4.3 PRODUCTS OF INCOMPLETE COMBUSTION EMISSIONS

The combustion of any organic compound results in the emission of generally low levels of incomplete combustion products. At the CRF VOST samples are routinely analyzed for 22 organic compounds using GC/FID. Tables 13 and 14 summarize the higher-concentration compounds found during these tests. The other nine compounds routinely analyzed for were not detected in all but at most two test samples.

Data noted for methylene chloride, 1,1-dichloroethylene, 1,2-dichloroethane, 1,1,1-trichloroethane, carbon tetrachloride, trichloroethylene, benzene, hexane, and toluene in Tables 13 and 14 may be compromised somewhat. After tests were performed the lot of pesticide grade methanol solvent used to clean VOST train component parts was found to be contaminated with low levels of constituents which chromatograph like those compounds noted above. The extent of data compromise is not known. However, as noted below, results noted in Tables 13 nd 14 compare well with past experience. In view of this, the results are felt to be valid.

At the afterburner exit, carbon tetrachloride (one of the POHCs) was present at the highest levels of all the compounds detected (generally >100 µg/dscm). Other chlorinated compounds such as methylene chloride, chloroform, and chlorinated alkanes and alkenes were present in the 10 to 100 µg/dscm range. Chlorobenzene concentrations were similar to those of the common chlorinated PICs.

At the scrubber exit, PIC concentrations are about one-tenth of those at the afterburner exit. Carbon tetrachloride levels ranged from 4.4 to 31 µg/dscm. The highest concentration occurred at a low excess air condition (test 9). Except for one test, chlorobenzene levels ranged from 1.4 to 14 µg/dscm and did not appear to correlate with temperature nor excess air. Other PICs include methylene chloride which was present in all tests at relatively high levels (11 to 139 µg/dscm). Chloroform was also present, at levels ranging from 5 to 74 µg/dscm. Other chlorinated alkanes and alkenes, hexane, benzene and toluene were present at levels generally below 10 µg/dscm.

TABLE 13. AFTERBURNER EXIT PIC DATA

| Test Number | 4 | 2 |
|---|---|---|
| Afterburner Exit $O_2$ (Percent) | 10.5 | 9.2 |
| Primary Combustor Temperature, °C (°F) | 1,307 (2,384) | 1,336 (2,437) |
| **POHC Concentration (µg/dscm)** | | |
| Carbon Tetrachloride | 110 | 150 |
| Chlorobenzene | 37 | 11 |
| **PIC Concentration (µg/dscm)** | | |
| Methylene Chloride | 18 | 77 |
| 1,1-Dichloroethylene | 38 | 6.7 |
| Chloroform | 70 | 12 |
| 1,2 Dichloroethane | 38 | 58 |
| 1,2-Dichloroethylene | 38 | 58 |
| 1,1,1 Trichloroethane | ND | ND |
| Trichloroethylene | 21 | 14 |
| Benzene | 13 | 3 |
| 1,1,2-Trichloroethane | 61 | 23 |
| Hexane | 15 | 10 |
| Toluene | 12 | 11 |

ND -- Not detected

TABLE 14. SCRUBBER DISCHARGE PIC DATA

| Test Number | 5 | 13 | 18 | 16 | 2 | 14 | 3 | 6 | 9 | 15 |
|---|---|---|---|---|---|---|---|---|---|---|
| Afterburner Exit $O_2$, (Percent) | 10.8 | 11.8 | 10.7 | 7.4 | 9.2 | 9.2 | 8.4 | 10.1 | 5.9 | 8.1 |
| Primary Combustor Temperature, °C (°F) | 1,137 (2,079) | 1,203 (2,198) | 1,176 (2,148) | 1,450 (2,642) | 1,336 (2,437) | 1,293 (2,359) | 1,228 (2,243) | 1,290 (2,354) | 1,425 (2,597) | 1,265 (2,310) |
| **POHC Concentration (µg/dscm)** | | | | | | | | | | |
| Carbon Tetrachloride | 4.4 | 7.5 | 12 | 23 | 12 | 26 | 15 | 20 | 31 | 9 |
| Chlorobenzene | 1.6 | 1.4 | 1.6 | 1.8 | 4.0 | 164 | 5 | 2 | 14 | 2 |
| **PIC Concentration (µg/dscm)** | | | | | | | | | | |
| Methylene Chloride | 15 | 34 | 42 | 67 | 18 | 139 | 14 | 20 | 11 | 14 |
| 1,1-Dichloroethylene | 0.6 | ND | ND | ND | ND | 0.5 | 4 | ND | ND | ND |
| Chloroform | 74 | 8.9 | 8.8 | 18 | 25 | 56.3 | 39 | ND | 5 | 6 |
| 1,2 Dichloroethane | ND | ND | ND | 2 | 31 | 30 | 4 | 5 | 47 | 9 |
| 1,2-Dichloroethylene | 8.7 | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 1,1,1-Trichloroethane | 0.2 | 0.8 | 0.6 | 1.0 | 1.3 | 2 | 1 | 2 | 1 | 1 |
| Trichloroethylene | 1.3 | 1.9 | 3.3 | 2.1 | 1.6 | 19 | 2.1 | 2 | 3 | 1 |
| Benzene | 2.6 | 1.3 | 1.6 | 1.0 | 6.3 | 12 | 28 | 2 | 2 | 2 |
| 1,1,2-Trichloroethane | 9.5 | 1.9 | 0.5 | 5.2 | ND | 10 | 9 | 4 | 2 | ND |
| Hexane | 0.9 | 1.9 | 5.5 | 2.0 | 2.0 | 21 | 2 | 2 | 1 | 1 |
| Toluene | 1.6 | 1.9 | 8.0 | 2.7 | 4.0 | 14 | 5 | 5 | 7 | 4 |

ND -- Not detected

## 4.4 PARTICULATE EMISSIONS

Table 15 summarizes the particulate levels measured at the afterburner exit, the scrubber exit, and in the stack. For the stack samples, the probe wash and filter particulate from the Method 5 train provided the typical Method 5 particulate loading data. For the afterburner exit and the scrubber exit samples, the probe wash samples were digested and analyzed for antimony and arsenic. Hence, the weights of the probe washes were not determined and were not included in the reported particulate concentrations.

At the afterburner exit, the particulate collected on the filter of the Method 5 train amounted to no more than 14 mg/dscm (corrected to 7 percent $O_2$). At the scrubber exit, between 5 and 53 mg/dscm were measured. At the stack, an average of 21 mg/dscm were measured with a highest concentration of 69 mg/dscm. All these particulate levels are below the hazardous waste incinerator performance standard of 180 mg/dscm (corrected to 7 percent $O_2$).

TABLE 15. PARTICULATE EMISSION SUMMARY

| | Afterburner exit particulate load[a] (mg/dscm) | | Scrubber exit particulate load[a] (mg/dscm) | | Stack Particulate load (mg/dscm) | |
|---|---|---|---|---|---|---|
| Test No. | As measured | Corrected to 7% $O_2$ | As measured | Corrected to 7% $O_2$ | As measured | Corrected to 7% $O_2$ |
| 2 | 12 | 14 | 41 | 48 | <1 | <1 |
| 3 | 6 | 7 | 12 | 17 | 8 | 12 |
| 4 | <1 | 1 | 23 | 36 | 11 | 18 |
| 5 | <1 | <1 | 7 | 15 | 7 | 15 |
| 6 | 3 | 4 | 10 | 14 | 8 | 12 |
| 7 | 2 | 2 | 52 | 53 | 18 | 18 |
| 9 | 6 | 6 | 4 | 5 | 10 | 12 |
| 13 | <1 | <1 | 8 | 16 | 6 | 11 |
| 14 | 12 | 13 | 21 | 33 | 14 | 22 |
| 15 | 3 | 3 | 4 | 7 | 15 | 25 |
| 16 | 11 | 12 | 38 | 53 | 49 | 69 |
| 18 | 5 | 7 | 8 | 15 | 9 | 16 |

[a]Does not include probe wash particulate; entire probe wash sample digested for antimony and arsenic analyses

# SECTION 5

# CONCLUSIONS

A series of pilot scale incineration tests were performed in the liquid injection incinerator system at EPA Combustion Research Facility to investigate possible dependences of volatile trace element emissions and scrubber blowdown discharge concentrations on incinerator operating conditions and synthetic waste feed chlorine content. The volatile trace elements investigated were arsenic and antimony. Specific test objectives were:

- To track the emissions of arsenic and antimony through the incinerator system
- To study the distribution of metals between ash and scrubber water
- To explore the relationship between metal emissions and incinerator operating conditions and feed chlorine content
- To evaluate whether the metals affect the destruction of other hazardous waste components
- To extend the data base on product of incomplete combustion (PIC) emissions from the incineration of chlorinated hydrocarbons

Twelve tests were performed with arsenic present in the synthetic waste fed at 1.9 to 14.5 ppm, and antimony present at 33 to 87 ppm. Test variables were incineration temperature, which was varied from 1,137° to 1,450°C (2,079° to 2,642°F); afterburner exit flue gas $O_2$, which was varied from 5.9 to 11.7 percent; and feed chlorine content, which was varied from 0 to 33.8 percent. The chlorine content of the feed was changed via the addition of varying amounts of carbon tetrachloride and chlorobenzene to a methanol base.

Major conclusions from the test results addressing each of the above objectives are:

- Antimony was relatively evenly distributed between the particulate and vapor phases in flue gas samples taken at the afterburner exit. The same was generally true for arsenic, although a greater fraction was found in the particulate phase for the greater number of tests.

Most, if not all, of the antimony and arsenic was found in the particulate catch of the sampling trains run at the scrubber exit.

- The success in achieving mass balance closure in the flue gas at the afterburner exit, and in the scrubber exit flue gas and scrubber blowdown discharge was in keeping with past experience. Mass balance closure was within a factor of 3 for 6 to 8 of the 12 tests performed for each element/location (afterburner exit or scrubber discharges) combination. Four of the 12 tests had very low (less than 2 percent) both antomony and arsenic recovery in the afterburner exit flue gas. The lowest recovery in scrubber discharges (flue gas plus scrubber blowdown) were better; 15 percent for antimony and 4 percent for arsenic. However, no account was taken for accumulation of the elements in the recirculating scrubber liquor. This may have affected the ability to achieve better mass balance closure.

- Comparable fractions of the input antimony were accounted for in the scrubber exxit flue gas and the scrubber blowdown. The same was largely true for arsenic, although a greater fraction of arsenic was found in the scrubber exit flue gas in a greater number of tests.

- No clear dependence of the distributions of arsenic and antimony discharges with the primary test variables was apparent. However, a statistical evaluation of the data obtained is planned in future efforts. This analysis will establish whether statistically significant relationships exist.

- Carbon tetrachloride DRE was greater than 99.99 percent as measured at the afterburner exit; this increased to greater than 99.999 percent as measured at the scrubber exit. Chlorobenzene DRF was greater than 99.999 at the afterburner exit, increasing to generally greater than 99.9999 percent at the scrubber exit. No clear dependence of DRE on incinerator operation was apparent. This DRE performance is consistent with past experience at the CRF, so the presence of the elements fed had no apparent effect on POHC DRE.

- Several commonly measured chlorinated $C_1$ and $C_2$ hydrocarbons, benzene, and toluene were present in the afterburner exit flue gas at levels in the 10 to several hundred µg/dscm range. These same compounds were generally present in the scrubber exit flue gas at levels about a factor 10 lower.

A major problem complicating efforts to perform a statistical evaluation of test data was the fact that actual test conditions deviated from target conditions, substantially in some instances. These deviations are largely due to the manual liquid feed, auxiliary fuel, and combustion air flowrate control system of the LIS. Plans are to upgrade this system to automatic feedback control, which will greatly facilitate setting and maintain future target conditions for system operation.

Data quality assurance (QA) objectives for measurement precision and accuracy were not fully met for many measurement parametes. However, failure to attain these had little impact on the above-stated conclusions.

# SECTION 6

# QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) procedures were performed to assess measurement precision and accuracy for four measurement parameters in the tests reported herein:

- Volatile organic priority pollutants (carbon tetrachloride and chlorobenzene) in organic liquid samples
- Volatile organic priority pollutants in flue gas
- Priority pollutant metals (arsenic and antimony) in aqueous samples (Method 5 train probe wash and impinger solutions)
- Priority pollutant metals (arsenic and antimony) in organic liquid samples

Both method and blind duplicate and spike (audit) samples were analyzed for the above parameters to assess measurement precision and accuracy, respectively. The QA objectives for each of these parameters as documented in the QA project plan for the CRF (Reference 7) are given in Table 16. Results of the duplicate and spike sample analyses are discussed in the following sections for each of the parameters challenged.

## 6.1 VOLATILE ORGANIC PRIORITY POLLUTANTS IN ORGANIC LIQUID SAMPLES

Each liquid synthetic waste feed formulation was prepared on a daily basis by combining measured weights of each of the three liquid components (methanol, carbon tetrachloride and chlorobenzene). If composite samples of these feeds, taken over the course of a given test, are considered audit spike samples, the analytical results for these measurements give a measure of method accuracy. Table 17 compares the analytical results for carbon tetrachloride and chlorobenzene with the blend preparation properties and notes method accuracy assuming the blend proportion result is the true value. As shown in the table, all carbon tetrachloride measurements except one met the QA objective for this measurement. In fact, except for this one result, analyzed values were within 56 percent of as prepared values. With one exception out of eleven analyses, the completeness objective was met.

TABLE 16. CRF QA OBJECTIVES FOR MEASUREMENTS PERFORMED IN THESE TESTS

| Measurement parameter | Method | QA objective | | |
|---|---|---|---|---|
| | | Precision (% RSD[a]) | Accuracy (%) | Completeness (%) |
| Volatile organic priority pollutants in organic liquid samples | Direct injection (dilution if needed) GC/MS by Method 8240 | 30 | -50 +100 | 90 |
| Volatile organic priority pollutants in flue gas | VOST followed by thermal desorption purge and trap GC/ECD by Method 8010 or GC/FID by Method 8020 | 30 | -50 +100 | 90 |
| Priority pollutant metals in aqueous samples | AA by 7000 series methods | 20 | ±30 | 90 |
| Priority pollutant metals in organic liquid samples | Acid digestion (Method 3030) with AA analysis by 7000 series methods | 20 | ±30 | 90 |

[a]Relative Standard Deviation

TABLE 17. METHOD ACCURACY FOR CARBON TETRACHLORIDE AND CHLOROBENZENE IN ORGANIC LIQUIDS

| Carbon tetrachloride | | | Chlorobenzene | | |
|---|---|---|---|---|---|
| Concentration (% wt) | | | Concentration (% wt) | | |
| As prepared | Analyzed | Accuracy | As prepared | Analyzed | Accuracy |
| 11.1 | 31.8 | +186 | 24.0 | 33.9 | +41 |
| 11.5 | 12.2 | +6 | 23.2 | 22.7 | -2 |
| 11.0 | 15.1 | +37 | 24.2 | 27.3 | +13 |
| 21.9 | 27.9 | +27 | 24.0 | 24.9 | +4 |
| 23.0 | 28.3 | +23 | 23.1 | 22.3 | -3 |
| 0 | -- | -- | 24.1 | 26.1 | +8 |
| 22.2 | 28.0 | +26 | 23.9 | 25.9 | +8 |
| 0 | -- | -- | 24.0 | 26.3 | +10 |
| 21.3 | 33.2 | +56 | 23.8 | 25.3 | +6 |
| 11.0 | 12.7 | +15 | 24.0 | 26.3 | +10 |
| 29.5 | 31.4 | +6 | 25.6 | 26.8 | +5 |
| 11.0 | 12.8 | +16 | 24.0 | 27.8 | +16 |
| 11.1 | 13.4 | +21 | 22.9 | 29.4 | +28 |
| Average | | +38 | | | +11 |
| Median | | +23 | | | +8 |

All chlorobenzene analyses were within the QA objective for this measurement. All 13 analyzed values were within 42 percent of as prepared values.

The QA objectives for this measurement were met.

## 6.2 VOLATILE ORGANIC PRIORITY POLLUTANTS IN FLUE GAS

Method accuracy for this parameter was measured by preparing field spike samples and analyzing these along with actual field samples. Table 18 summarizes the results of the field spike VOST trap pair analyses. Table 19 summarizes the percentage of spike sample analyses which met the measurement accuracy objective of -50, +100 percent. As noted in Table 19, greater than 90 percent of the spike sample analyses met the accuracy objective for 19 of the 22 compounds analyzed. The three compounds failing 90 percent within the accuracy objective were carbon tetrachloride, bromodichloromethane, and t-1,3-dichloropropene. No data for bromodichloromethane nor t-1,2-dichloropropane were reported in Section 4.3, so failure to meet the accuracy objective for these measurements has no bearing on test conclusions.

Unfortunately, carbon tetrachloride was one of the designated POHCs for these tests. Three of 11 spike sample analyses were out of accuracy bounds for its measurement. In all three of these, the measured value was higher than the spiked value. If, then, carbon tetrachloride analyses bias toward measuring greater amounts than are present, reported levels in Section 4.3 may be greater than actual. The test conclusion was that greater than 99.99 percent carbon tetrachloride DRE was achieved at the afterburner exit, increasing to greater than 99.999 percent at the scrubber discharge. This conclusion may be slightly conservative.

Overall, 229 individual analysis results out of 242 individual compound analyses in spike samples met the measurement QA objective, an overall completeness of 95 percent.

In accordance with method procedures, surrogates are injected onto each VOST trap pair analyzed, and surrogate recovery measured. Surrogate recoveries for all sample, field blank, and field spike samples reported in this report are given in Table 20.

Although no QA objective for surrogate recovery is stated in the project QA plan, the appropriate one would be that for accuracy of the volatile organic priority pollutants in flue gas. As noted in Table 16, this objective is -50, +100 percent accuracy. When translated to percent recovery, the appropriate objective would be 50 to 200 percent recovery.

The data in Table 20 show that isooctane recovery was between 50 to 200 percent for 51 of 65 trap pairs analyzed, or 78 percent. For octane, surrogate recovery was between 50 and 200 percent for 57 of 65 trap pairs analyzed, or 88 percent. Both of these would fail an implied completeness objective of 90 percent, although octane recovery just barely.

TABLE 18. MEASUREMENT ACCURACY FOR VOLATILE ORGANIC PRIORITY POLLUTANTS IN FLUE GAS: VOST SPIKE SAMPLE ANALYSIS RESULTS

| Compound | Quantitation limit (ng/trap pair) | Spike level (ng/trap pair) | 001211345VFSK | | 001271348VFSK | | 001291038VFSK | | 002101226VFSK | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) |
| Methylene chloride | 63.0 | 630 | 591 | -6 | 887 | 41 | 655 | 4 | 702 | 11 |
| 1,1-Dichloroethylene | 12.6 | 126 | 106 | -16 | 172 | 37 | 132 | 5 | 150 | 19 |
| 1,1-Dichloroethane | 12.6 | 126 | 106 | -16 | 159 | 26 | 142 | 13 | 140 | 11 |
| t-1,2-Dichloroethylene | 12.6 | 126 | 104 | -17 | 161 | 28 | 139 | 10 | 143 | 13 |
| Chloroform | 21.1 | 211 | 377 | 79 | 287 | 36 | 257 | 22 | 215 | 2 |
| 1,2-Dichloroethane | 25.3 | 253 | 312 | 23 | 274 | 8 | 275 | 9 | 276 | 9 |
| 1,1,1-Trichloroethane | 12.6 | 126 | 152 | 21 | 156 | 24 | 150 | 19 | 137 | 9 |
| Carbon tetrachloride | 12.6 | 126 | 682 | 440 | 172 | 37 | 907 | 620 | 153 | 21 |
| Bromodichloromethane | 21.1 | 211 | <21 | -90[a] | 222 | 5 | 318 | 51 | 214 | 1 |
| 1,2-Dichloropropane | 12.6 | 126 | 112 | -11 | 138 | 10 | 137 | 9 | 130 | 3 |
| t-1,3-Dichloropropane | 25.2 | 252 | 196 | -22 | 254 | 1 | 275 | 9 | 251 | 0 |
| Trichloroethylene | 12.6 | 126 | 160 | 27 | 141 | 12 | 137 | 9 | 138 | 10 |
| Benzene | 12.7 | 127 | 169 | 33 | 146 | 15 | 165 | 30 | 137 | 8 |
| 1,1,2-Trichloroethane | 33.7 | 337 | 259 | -23 | 336 | 0 | 374 | 11 | 316 | -6 |
| Hexane | 12.7 | 127 | 558 | 340 | 173 | 36 | 166 | 31 | 176 | 39 |
| Bromoform | 21.1 | 211 | 136 | -36 | 205 | -3 | 165 | -22 | 224 | 6 |
| Tetrachloroethylene +Tetrachloroethane | 25.2 | 252 | 207 | -18 | 260 | 3 | 262 | 4 | 268 | 6 |
| Toluene | 25.4 | 254 | 258 | 2 | 288 | 13 | 291 | 15 | 270 | 6 |
| Chlorobenzene | 63.0 | 630 | 539 | -14 | 651 | 3 | 700 | 11 | 650 | 3 |
| 1,3-Dichlorobenzene | 63.0 | 630 | 566 | -10 | 579 | -8 | 483 | -23 | <63 | -90[a] |
| 1,2-Dichlorobenzene | 63.0 | 630 | 545 | -13 | 568 | -10 | 518 | -18 | <63 | -90[a] |
| 1,4-Dichlorobenzene | 63.0 | 630 | 544 | -14 | 568 | -10 | 479 | -24 | <63 | -90[a] |

[a]Assumed present at the detection limit for accuracy calculation.

TABLE 18. MEASUREMENT ACCURACY FOR VOLATILE ORGANIC PRIORITY POLLUTANTS IN FLUE GAS: VOST SPIKE SAMPLE ANALYSIS RESULTS (CONTINUED)

| Compound | Quantitation limit (ng/trap pair) | Spike level (ng/trap pair) | 002121215VFSK | | 002131104VFSK | | 002181247VFSK | | 002191002VFSK | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) |
| Methylene chloride | 63.0 | 630 | 494 | -22 | 833 | 32 | 581 | -8 | 686 | 9 |
| 1,1-Dichloroethylene | 12.6 | 126 | 97 | -23 | 164 | 30 | 168 | 33 | 110 | -13 |
| 1,1-Dichloroethane | 12.6 | 126 | 98 | 22 | 152 | 21 | 97 | -23 | 123 | -2 |
| t-1,2-Dichloroethylene | 12.6 | 126 | 98 | -22 | 157 | 25 | 117 | -7 | 111 | -12 |
| Chloroform | 21.1 | 211 | 175 | -17 | 234 | 11 | 214 | 1 | 220 | 4 |
| 1,2-Dichloroethane | 25.3 | 253 | 206 | -19 | 297 | 17 | 329 | 30 | 209 | -17 |
| 1,1,1-Trichloroethane | 12.6 | 126 | 102 | -19 | 150 | 19 | 49 | -61 | 130 | 3 |
| Carbon tetrachloride | 12.6 | 126 | 105 | -17 | 170 | 35 | 80 | -37 | 290 | 130 |
| Bromodichloromethane | 21.1 | 211 | 180 | -15 | 228 | 8 | <21 | -90[a] | 237 | 12 |
| 1,2-Dichloropropane | 12.6 | 126 | 102 | -19 | 135 | 7 | 96 | -24 | 122 | -3 |
| t-1,3-Dichloropropane | 25.2 | 252 | 193 | -23 | 233 | -8 | 64 | -75 | 406 | 61 |
| Trichloroethylene | 12.6 | 126 | 103 | -18 | 142 | 13 | 126 | 0 | 118 | -6 |
| Benzene | 12.7 | 127 | 110 | -13 | 164 | 30 | 129 | 2 | 121 | -5 |
| 1,1,2-Trichloroethane | 33.7 | 337 | 258 | -23 | 306 | -9 | 252 | -25 | 427 | 27 |
| Hexane | 12.7 | 127 | 101 | -20 | 176 | 39 | 127 | 0 | 122 | -4 |
| Bromoform | 21.1 | 211 | 180 | -15 | 201 | -5 | 152 | -28 | 210 | 0 |
| Tetrachloroethylene +Tetrachloroethane | 25.2 | 252 | 241 | -4 | 255 | 1 | 250 | -1 | 227 | -10 |
| Toluene | 25.4 | 254 | 218 | -14 | 283 | 11 | 232 | -9 | 233 | -8 |
| Chlorobenzene | 63.0 | 630 | 559 | -11 | 616 | -2 | 824 | 31 | 665 | 6 |
| 1,3-Dichlorobenzene | 63.0 | 630 | 515 | -18 | 591 | -6 | 588 | -7 | 552 | -12 |
| 1,2-Dichlorobenzene | 63.0 | 630 | 540 | -14 | 589 | -7 | 654 | 4 | 570 | -10 |
| 1,4-Dichlorobenzene | 63.0 | 630 | 526 | -17 | 593 | -6 | 609 | -3 | 557 | -12 |

[a]Assumed present at the detection limit for accuracy calculation.

TABLE 18. MEASUREMENT ACCURACY FOR VOLATILE ORGANIC PRIORITY POLLUTANTS IN FLUE GAS: VOST SPIKE SAMPLE ANALYSIS RESULTS (CONCLUDED)

| Compound | Quantitation limit (ng/trap pair) | Spike level (ng/trap pair) | 002201255VFSK | | 002241021VFSK | | 002261120VFSK | |
|---|---|---|---|---|---|---|---|---|
| | | | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) | Concentration (ng/trap pair) | Accuracy (%) |
| Methylene chloride | 63.0 | 630 | 566 | -10 | 643 | 2 | 729 | 15 |
| 1,1-Dichloroethylene | 12.6 | 126 | 154 | 22 | 86 | -32 | 131 | 4 |
| 1,1-Dichloroethane | 12.6 | 126 | 94 | -25 | 112 | -11 | 138 | 10 |
| t-1,2-Dichloroethylene | 12.6 | 126 | 110 | -13 | 109 | -13 | 141 | 12 |
| Chloroform | 21.1 | 211 | 178 | -16 | 195 | -8 | 241 | 14 |
| 1,2-Dichloroethane | 25.3 | 253 | 197 | -22 | 270 | 7 | 245 | -3 |
| 1,1,1-Trichloroethane | 12.6 | 126 | 86 | -32 | 145 | 15 | 162 | 29 |
| Carbon tetrachloride | 12.6 | 126 | 99 | -21 | 166 | 32 | 191 | 52 |
| Bromodichloromethane | 21.1 | 211 | 150 | -29 | 196 | -7 | 237 | 12 |
| 1,2-Dichloropropane | 12.6 | 126 | 78 | -38 | 116 | -8 | 128 | 2 |
| t-1,3-Dichloropropane | 25.2 | 252 | 77 | -69 | 390 | 55 | 348 | 38 |
| Trichloroethylene | 12.6 | 126 | 168 | 33 | 127 | 1 | 112 | -11 |
| Benzene | 12.7 | 127 | 112 | -12 | 115 | -9 | 141 | 11 |
| 1,1,2-Trichloroethane | 33.7 | 337 | 156 | -54 | 402 | 19 | 373 | 11 |
| Hexane | 12.7 | 127 | 109 | -14 | 125 | -2 | 156 | 23 |
| Bromoform | 21.1 | 211 | <21 | -90# | 216 | 2 | 188 | -11 |
| Tetrachloroethylene +Tetrachloroethane | 25.2 | 252 | 433 | 72 | 245 | -3 | 283 | 12 |
| Toluene | 25.4 | 254 | 291 | 15 | 252 | 1 | 290 | 14 |
| Chlorobenzene | 63.0 | 630 | 608 | -3 | 692 | 10 | 694 | 10 |
| 1,3-Dichlorobenzene | 63.0 | 630 | 402 | -36 | 539 | -14 | 678 | 8 |
| 1,2-Dichlorobenzene | 63.0 | 630 | 341 | -46 | 567 | -10 | 672 | 7 |
| 1,4-Dichlorobenzene | 63.0 | 630 | 353 | -44 | 541 | -14 | 682 | 8 |

#Assumed present at the detection limit for accuracy calculation.

TABLE 19. INDIVIDUAL COMPOUND SPIKE SAMPLE ANALYSIS COMPLETENESS

| Compound | Percent of spike sample analyses meeting QA objective (-50, +100 percent) |
|---|---|
| Methylene chloride | 100 |
| 1,1-Dichloroethylene | 100 |
| 1,1-Dichloroethane | 100 |
| t-1,2-Dichloroethylene | 100 |
| Chloroform | 100 |
| 1,2-Dichloroethane | 100 |
| 1,1,1-Trichloroethane | 91 |
| Carbon tetrachloride | 73 |
| Bromodichloromethane | 82 |
| 1,2-Dichloropropane | 100 |
| t-1,3-Dichloropropene | 82 |
| Trichloroethylene | 100 |
| Benzene | 100 |
| 1,1,2-Trichloroethane | 100 |
| Hexane | 91 |
| Bromoform | 91 |
| Tetrachloroethylene + Tetrachloroethane | 100 |
| Toluene | 100 |
| Chlorobenzene | 100 |
| 1,3-Dichlorobenzene | 91 |
| 1,2-Dichlorobenzene | 91 |
| 1,4-Dichlorobenzene | 91 |
| Overall (all compounds) | 95 |

TABLE 20. SURROGATE RECOVERY FOR VOST ANALYSES

| Sample | Surrogate recovery (%) | | Sample | Surrogate recovery (%) | |
|---|---|---|---|---|---|
| | Isooctane | Octane | | Isooctane | Octane |
| A01211355V | 106 | 120 | Q02120958VFBK | 76 | 138 |
| A01211505V | 226 | 211 | Q02121215VFSK | 86 | 83 |
| A01211617V | 146 | 186 | E02130902V | 0 | 109 |
| Q01211345VFSK | 87 | 88 | E02131009V | 115 | 195 |
| Q01211600VFBK | 123 | 138 | E02131104V | 116 | 179 |
| A01271320V | 26 | 206 | Q02130955VFBK | 87 | 157 |
| A01271426V | 130 | 123 | Q02131104VFSK | 162 | 144 |
| A01271539V | 77 | 154 | E02181021V | 0 | 81 |
| A01271602V | 91 | 156 | E02181125V | 101 | 165 |
| E01271434V | 136 | 430 | E02181257V | 71 | 142 |
| Q01271348VFSK | 143 | 115 | Q02181022VFBK | 13 | 0 |
| Q01271513VFBK | 95 | 148 | Q02181257VFSK | 89 | 91 |
| A01291037V | 43 | 139 | E02191002V | 0 | 75 |
| A01291121V | 103 | 177 | E02191100V | 89 | 146 |
| A01291347V | 110 | 182 | E02191201V | 88 | 153 |
| Q01291038VFSK | 110 | 110 | Q02191002VFSK | 90 | 91 |
| Q01291149VFBK | 104 | 178 | Q02191201VFBK | 93 | 136 |
| E02031353V | 30 | 93 | E02201024V | 14 | 130 |
| E02031443V | 146 | 164 | E02201200V | 83 | 141 |
| E02031548V | 131 | 281 | E02201255V | 83 | 140 |
| Q02031548VFBK | 198 | 253 | Q02201024VFBK | 110 | 150 |
| E02051441V | 63 | 142 | Q02201255VFSK | 123 | 84 |
| E02051525V | 69 | 133 | E02241021V | 0 | 60 |
| E02051605V | 70 | 160 | E02241128V | 48 | 218 |
| E02101024V | 22 | 48 | E02241230V | 132 | 172 |
| E02101102V | 74 | 136 | Q02241021VFSK | 88 | 94 |
| E02101200V | 87 | 148 | Q02241230VFBK | 74 | 145 |
| E02101232V | 105 | 180 | E02260918V | 12 | 57 |
| Q02101109VFBK | 125 | 170 | E02261030V | 92 | 175 |
| Q02101226VFSK | 107 | 98 | E02261120V | 79 | 171 |
| E02120955V | 21 | 159 | Q02260918VFBK | 72 | 153 |
| E02121120V | 75 | 156 | Q02261120VFSK | 112 | 112 |
| E02121215V | 68 | 139 | | | |
| | | | Average | 87 | 149 |
| | | | Median | 87 | 146 |

An interesting pattern exists in the data for isooctane recovery. The sample numbers shown in the table code the type of sample and the date and time of its collection. The prefix "A" denotes a sample taken at the afterburner exit, "E" a sample taken in the scrubber system discharge, and "Q" a QA sample (blank or spike) prepared in the laboratory. The suffix "V" denotes a VOST sample, "VFSK" a VOST field spike sample, and "VFBK" a VOST field blank sample. The first four digits following the prefix denote the date the samples were taken; the next four digits correspond to the 24-hour clock time the sample was taken. Thus sample A01211355V would be a VOST sample of the afterburner exit flue gas taken at 1355 on January 21.

Samples are analyzed in the laboratory generally in the order prepared. By examining the isooctane surrogate recoveries for flue gas samples (suffix "V" only) for January 27 and 29, and February 3, 10, 12, 13, 18, 19, 20, 24, and 26 (11 out of 13 days during which VOST samples were analyzed) one notices that recovery is very poor (often 0) for the first trap pair analyzed each day, but is acceptable for succeeding trap pairs analyzed that day. The pattern is sufficiently repeatable to be significant. The source of this poor initial analysis isooctane recovery will be investigated.

Failure of an implied QA objective for surrogate recovery again does not affect test conclusions measurably. DRE results are order of magnitude in nature, as are PIC emission conclusions. Failure to strictly achieve a factor of 2 surrogate recovery objective with 90 percent completeness will not measurably affect order of magnitude conclusions.

## 6.3 PRIORITY POLLUTANT METALS IN AQUEOUS LIQUID SAMPLES

### 6.3.1 Measurement Accuracy

The accuracy of this parameter was measured by submitting blind spike samples for analysis. Blind spike samples labeled as scrubber blowdown water and as sampling train impinger solutions were submitted. Table 21 summarizes the QA data obtained for this measurement. As shown in Table 21, 18 blind audit samples were submitted for analysis; two of these were analyzed in duplicate under the laboratory's standard QA procedure. In addition, the laboratory prepared five additional spike samples in accordance with their standard QA procedures. Thus, 25 audit sample analyses were performed.

For the arsenic analyses, all impinger solution spike samples (those beginning with the letter E) were analyzed with accuracy better than -33 to +20 percent. Twenty of the 21 impinger spikes had accuracy within the QA objective of ±30 percent. The accuracy for the three blowdown sample analyses (those samples with the prefix B in Table 21) was poorer, ranging from +46 to +67 percent. In all, 20 of 24 results were within the accuracy objective, for a completeness of 83 percent. This just fails the QA objective of 90 percent completeness for this measurement.

As noted above, only blowdown water spike samples failed the accuracy objective. Spike analysis results, thus, suggest that the blowdown water sample analyses might be in error by up to a factor of 2. However, in view

TABLE 21. MEASUREMENT ACCURACY FOR ARSENIC AND ANTIMONY DETERMINATIONS IN AQUEOUS LIQUID SAMPLES

| Sample description | Arsenic Concentration (mg/L) Spiked | Arsenic Concentration (mg/L) Analyzed | Arsenic Accuracy (%) | Antimony Concentration (mg/L) Spiked | Antimony Concentration (mg/L) Analyzed | Antimony Accuracy (%) |
|---|---|---|---|---|---|---|
| Blind Spike Samples | | | | | | |
| B01301116 | 0.310 | 0.49 | +58 | 1.90 | 1.7 | -11 |
| B02051246 | 0.144 | 0.24 | +67 | 2.50 | 3.2 | +28 |
| B02260915 | 0.144 | 0.21 | +46 | 2.50 | 2.7 | +8 |
| E0116120112 | 0.036 | 0.04 | +11 | 57.5 | 40 | -30 |
| E0121130013 | 0.048 | 0.05 | +4 | 42.5 | 31 | -27 |
| E0127120014 | 0.060 | 0.06 | +0 | 34.5 | 24 | -30 |
| E0129101012 | 0.072 | 0.07 | -3 | 25.5 | 21 | -18 |
| E0130110012 | 0.084 | 0.07 | -17 | 11.5 | 9.8 | -15 |
| E0203104014 | 0.096 | 0.07 | -27 | 8.50 | 6.9 | -19 |
| E0205122013 | 0.108 | 0.08 | -26 | 4.60 | 3.1 | -33 |
| E0210095812 | 0.132 | 0.10 | -24 | 3.40 | 2.4 | -29 |
| E0212100013 | 0.192 | 0.15 | -22 | 1.15 | 1.2 | +4 |
| E0213090014 | 0.267 | 0.18 | -33 | 0.850 | 0.85 | +0 |
| E0218160013 | 0.324 | 0.35 | +8 | 0.115 | 0.11 | -4 |
| E0219153014 | 0.396 | 0.40 | +10 | 0.085 | 0.086 | +1 |
| E0222164013 | 0.444 | 0.40 | -10 | 0.023 | 0.026 | +13 |
| E0224154512 | 0.528 | 0.63 | +19 | 0.017 | 0.024 | +11 |
| E0226162114 | 0 | 0.02 | -- | 100 | 55 | -45 |
| Blind Spike Samples Analyzed in Duplicate | | | | | | |
| E0129101012 | 0.072 | 0.07 | -3 | 25.5 | 23 | -10 |
| E0224154512 | 0.528 | 0.52 | -2 | 0.017 | 0.027 | +59 |
| Laboratory Spike Samples | | | | | | |
| E0130120311 | -- | 75[a] | -25 | -- | 100[a] | +0 |
| E02051405PW | -- | 78[a] | -22 | -- | 72[a] | -28 |
| E0212100013 | -- | 131[a] | +31 | -- | SL[b] | -- |
| E0213085011 | -- | 128[a] | +28 | -- | 72[a] | -28 |
| E0213085013 | -- | 78[a] | -22 | -- | 78[a] | -22 |
| Average | | | 0 | | | -9 |
| Median | | | 0 | | | -10 |

[a]Result reported as percent recovery.
[b]Spike level too low when compared to background concentration.

of the scatter in the data noted in Section 4, errors of this magnitude would have little bearing on test conclusions.

For the antimony analyses, 20 of 24 audit samples analyzed had an accuracy of between -30 and +30 percent, for a completeness again of 83 percent. This just fails the completeness objective for this parameter of 90 percent. Still, as noted above, given the high variability in reported test results, borderline failure of this parameter's QA objective is deemed to have little effect on test conclusions.

### 6.3.2 Measurement Precision

The precision of this measurement was evaluated by submitting blind duplicate samples to the laboratory for analysis. In addition, the laboratory also analyzed some samples in duplicate as part of the laboratory's internal QA program. Results of these analyses are summarized in Table 22.

As shown in Table 22, 18 of 22 samples met the QA precision objective of 20 percent RSD for the arsenic anlaysis, or 82 percent of samples analyzed. The four samples failing, again, were blowdown water samples. These failures are expected to have no bearing on test conclusions for the reasons noted above.

Nineteen of 22 samples met the QA precision objective for the antimony analysis, or 86 percent. Again, the borderline failure (just under 90 percent acceptable results) of the QA objective is expected to have no bearing on test conclusions.

## 6.4 PRIORITY POLLUTANT METALS IN ORGANIC LIQUID SAMPLES

### 6.4.1 Measurement Accuracy

Four blind audit samples of liquid feed mixtures containing known amounts of arsenic and antimony were submitted for analysis. Results are shown in Table 23. As shown, measurement accuracy for arsenic ranged from +63 to +250 percent, well out of bounds of the QA objective for this measurement of $\pm$30 percent. For antimony, measurement accuracy ranged from +23 to +100 percent. Only one of the four determinations met the QA objective.

Normally such a failure to achieve the accuracy objective for such a measurement would be expected to compromise test results. However, as noted in Section 4.1, test results were erractic and mass balance closure, though within the range seen in past efforts to close a mass balance around a combustion source, was poor. As a consequence, the basic test conclusion was that no clear dependence between arsenic/antimony discharge distributions and primary test variables was evident. Failure to achieve the QA accuracy objective for arsenic and antimony analyses in feed samples would not alter this conclusion.

TABLE 22. MEASUREMENT PRECISION FOR ARSENIC AND ANTIMONY DETERMINATIONS IN AQUEOUS LIQUID SAMPLES

| Sample primary description | Arsenic Concentration (mg/L) | | Arsenic Precision (% RSD[a]) | Antimony Concentration (mg/L) | | Antimony Precision (% RSD[a]) |
|---|---|---|---|---|---|---|
| | Meas. 1 | Meas. 2 | | Meas. 1 | Meas. 2 | |
| **Blind Duplicates Submitted** | | | | | | |
| B01271350 | 0.015 | <0.01 | 28[b] | 0.72 | 0.54 | 20 |
| B02121000 | 0.02 | 0.05 | 61 | 0.50 | 0.51 | 1 |
| B02181000 | 0.04 | 0.04 | 0 | 0.78 | 0.76 | 2 |
| B02191000 | 0.08 | 0.05 | 33 | 0.75 | 0.77 | 2 |
| B02241100 | 0.02 | <0.01 | 47[b] | 0.72 | 0.69 | 3 |
| **Laboratory Duplicates** | | | | | | |
| AB0116102011 | <0.005 | <0.005 | 0 | 0.026 | 0.031 | 12 |
| AB0127135012 | 0.013 | 0.013 | 0 | 2.7 | 3.4 | 16 |
| A0212100011 | 1.0 | 1.7 | 8 | 12 | 9.9 | 14 |
| A02260900PW | 1.0 | 0.88 | 9 | 7.5 | 8.2 | 6 |
| B01271350 | <0.01 | <0.01 | 0 | 0.54 | 0.54 | 0 |
| B02051245 | 0.03 | 0.04 | 20 | 0.83 | 0.84 | 1 |
| E0127132714 | <0.005 | <0.005 | 0 | 0.108 | 0.090 | 13 |
| E0129101012 | 0.07 | 0.07 | 0 | 21 | 23 | 6 |
| E0203133512 | <0.005 | <0.005 | 0 | 0.011 | 0.007 | 31 |
| E0205140511 | <0.01 | <0.01 | 0 | 0.021 | 0.023 | 6 |
| E0205140514 | <0.01 | <0.01 | 0 | 0.046 | 0.049 | 4 |
| E0210102011 | <0.01 | <0.01 | 0 | <0.005 | <0.005 | 0 |
| E0210102013 | <0.01 | <0.01 | 0 | 0.011 | 0.011 | 0 |
| E02190940PW | <0.005 | <0.005 | 0 | 0.010 | 0.015 | 28 |
| E02241010PW | <0.005 | <0.005 | 0 | <0.005 | 0.01 | 47[b] |
| E0224154512 | 0.63 | 0.52 | 14 | 0.024 | 0.027 | 8 |
| E0226090011 | <0.01 | <0.01 | 0 | 0.54 | 0.54 | 0 |
| Average | | | 10 | | | 10 |
| Median | | | 0 | | | 6 |

[a]Relative Standard Deviation
[b]RSD calculated assuming not detected present at the detection limit.

TABLE 23. MEASUREMENT ACCURACY FOR ARSENIC AND ANTIMONY DETERMINATIONS IN ORGANIC LIQUID SAMPLES

| Sample description | Arsenic | | | Antimony | | |
|---|---|---|---|---|---|---|
| | Concentration (mg/L) | | Accuracy (%) | Concentration (mg/L) | | Accuracy (%) |
| | Spiked | Analyzed | | Spiked | Analyzed | |
| Blind Spike Samples | | | | | | |
| F01161202 | 0.2 | 0.7 | +250 | 4.0 | 4.9 | +22 |
| F02051330 | 0.8 | 1.3 | +63 | 1.0 | 1.9 | +90 |
| F02221240 | 0.6 | 1.1 | +83 | 2.0 | 4.0 | +100 |
| F02261040 | 0.4 | 0.7 | +75 | 3.0 | 5.1 | +70 |
| Average | | | +118 | | | 70 |

6.4.2 Measurement Precision

Three blind duplicate samples of liquid feed were submitted for analysis. Results are shown in Table 24. For arsenic, the precision QA objective was met for all three. For antimony, this objective was not strictly met for all three, however the magnitude of failure was quite small.

TABLE 24. MEASUREMENT PRECISION FOR ARSENIC AND ANTIMONY DETERMINATIONS IN ORGANIC LIQUID SAMPLES

| Sample primary description | Arsenic | | | Antimony | | |
|---|---|---|---|---|---|---|
| | Concentration (mg/L) | | Precision (% RSD)[a] | Concentration (mg/L) | | Precision (% RSD)[a] |
| | Meas. 1 | Meas. 2 | | Meas. 1 | Meas. 2 | |
| Blind Duplicates | | | | | | |
| F01211321 | 1.6 | 1.9 | 12 | 30 | 42 | 24 |
| F01301111 | 15 | 14 | 5 | 73 | 54 | 21 |
| F02190901 | 2.7 | 3.3 | 14 | 39 | 27 | 26 |
| Average | | | 10 | | | 24 |

[a]Relative Standard Deviation

## REFERENCES


1. "Proposed Protocol for the CRF Testing of RCRA Metal Contaminated Synthetic Wastes," Acurex report prepared under EPA Contract 68-03-3267, December 1986.

2. Chemical Engineer's Handbook, R. H. Perry and C. H. Chilton, eds., McGraw-Hill, New York, 1973, p. 2-74.

3. "Test Methods of Evaluating Solid Waste: Physical/Chemical Methods," EPA SW-846, 2nd ed., July 1982.

4. J. C. Harris, et al., "Sampling and Analysis Methods for Hazardous Waste Incineration," EPA-600/8-84-002, February 1984.

5. 40 CFR Part 60, Appendix A.

6. E. M. Hansen, "Protocol for the Collection and Analysis of Volatile POHCs Using VOST," EPA-600/8-84-007, March 1984.

7. "Quality Assurance Project Plan for the Operation of the Combustion Research Facility," Acurex Corporation, prepared under EPA Contract 68-03-3267, March 1987.